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Bruna Dias Ilha

**DESAFIOS EROSIVOS E O USO DE DENTIFRÍCIO CONTENDO
SnCl₂: IMPLICAÇÕES NA RESISTÊNCIA DE UNIÃO DE UM ADESIVO
UNIVERSAL AO ESMALTE E À DENTINA**

Santa Maria, RS
2021

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ESMALTE E À DENTINA**

Tese apresentada ao Curso de Doutorado do Programa de Pós-Graduação em Ciências odontológicas, Área de Concentração em Odontologia, ênfase em Materiais Dentários, da Universidade Federal de Santa Maria (UFSM, RS) como requisito parcial para obtenção do grau de **Doutor em Ciências Odontológicas**.

Orientador: Prof. Dr. Fábio Zovico Maxnuck Soares

Santa Maria, RS
2021

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Aprovada em 7 de Outubro de 2021:



Fábio Zovico Maxnuck Soares, Dr. (UFSM)
(Presidente da banca/Orientador)



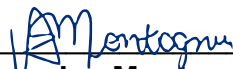
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RESUMO

DESAFIOS EROSIVOS E O USO DE DENTIFRÍCIO CONTENDO SnCl_2 : IMPLICAÇÕES NA RESISTÊNCIA DE UNIÃO DE UM ADESIVO UNIVERSAL AO ESMALTE E À DENTINA

AUTOR: Bruna Dias Ilha
ORIENTADOR: Fábio Zovico Maxnuck Soares

No presente trabalho, são apresentados dois artigos envolvendo o estudo do efeito do desafio erosivo, bem como do uso de dentifrício anti-erosivo, na resistência de união-(RU) e na estabilidade da adesão de um adesivo universal - Scotchbond Universal-(SBU) ao esmalte e à dentina. O primeiro, objetivou investigar a influência de um dentifrício contendo SnCl_2 em dentes com lesões de erosão na RU do SBU ao esmalte e à dentina. 120 incisivos bovinos foram divididos aleatoriamente em 12 grupos-(n=10) conforme: a) substrato: esmalte/dentina; b) condição do substrato: hígido/erodido (imersão em refrigerante de cola)/erodido-tratado (imersão em refrigerante de cola-aplicação de dentifrício); c) estratégia de aplicação-SBU: com condicionamento ácido-(ER)/autocondicionante-(SE). Foram confeccionados 4 cilindros de resina composta (0,96mm-diâmetro/1mm-altura) em cada face vestibular planificada. 2 cilindros foram testados em cada tempo de avaliação. Para avaliação da RU, o teste de microcislamento foi realizado imediatamente após 24h e 6 meses de armazenamento (água destilada, 37°C). Os valores de RU-(Mpa) foram analisados utilizando ANOVA-três fatores. Em dentina, houve interação significativa entre os três fatores observados ($P=0,023$). A dentina erodida reduziu os valores de RU-(24h) em ER. O tratamento com dentifrício também reduziu os valores de RU à dentina, independentemente da estratégia utilizada. Em esmalte, não houve interação significativa entre os fatores. Os fatores isolados condição do substrato ($P=0,026$), estratégia de aplicação ($P=0,004$) e tempo de armazenamento ($P=0,016$) foram significantes. O esmalte erodido não impactou na RU, comparado ao hígido. O desafio erosivo impacta negativamente na RU do SBU, em dentina, utilizado na estratégia ER enquanto o tratamento com dentifrício anti-erosivo compromete a adesão à dentina em ambas estratégias. Tanto o desafio erosivo quanto o tratamento não prejudicam a adesão ao esmalte. O segundo artigo objetivou avaliar o efeito do desafio erosivo após os procedimentos adesivos e restauradores em esmalte e dentina, na estabilidade da RU do SBU aplicado nas duas estratégias de condicionamento. 80 incisivos bovinos foram divididos aleatoriamente em 8 grupos-(n=10) conforme: a) substrato: esmalte/dentina; b) estratégia de aplicação-SBU: ER/SE; c) desafio após restauração: desafio erosivo em refrigerante de cola/armazenamento em saliva artificial. Foram confeccionados 4 cilindros de resina composta em cada superfície planificada, 2 foram submetidos ao teste de microcislamento para determinar a RU imediatamente após o desafio erosivo e o armazenamento em saliva artificial por 7 dias e 2 após 6 meses de armazenamento (água destilada, 37°C). Os valores de RU-(Mpa) foram analisados utilizando ANOVA-três fatores. Os fatores isolados desafio após restauração, estratégia de aplicação e tempo de armazenamento foram estatisticamente significantes ($P=0,000$, $P=0,048$ e $P=0,000$, respectivamente). Os valores de RU em dentina erodida foram reduzidos em ER. A estabilidade da RU à dentina foi afetada no grupo controle. No entanto, menores valores de RU imediata foram encontrados no grupo erodido. Em esmalte, foi observada significância estatística para o fator isolado estratégia de aplicação ($P=0,00$), os valores de RU em ER foram maiores do que em SE. O desafio erosivo após os procedimentos restauradores não compromete a estabilidade da adesão ao esmalte e à dentina; entretanto, prejudica a RU imediata à dentina, quando utilizada a estratégia ER.

Palavras-Chave: Colagem Dentária. Erosão Dentária. Resistência ao Cislamento.

ABSTRACT

EROSIVE CHALLENGES AND THE USE OF SnCl₂ CONTAINING TOOTHPASTE: IMPLICATIONS ON THE BOND STRENGTH OF A UNIVERSAL ADHESIVE TO ENAMEL AND DENTIN

AUTOR: Bruna Dias Ilha
ORIENTADOR: Fábio Zovico Maxnuck Soares

In the present study, two papers are presented involving the study of the effect of erosive challenge, as well as the use of anti-erosive dentifrice, on bond strength (μ SBS) and on the adhesion stability of a universal adhesive - Scotchbond Universal-(SBU) to enamel and dentin. The first article aimed to investigate the influence of a SnCl₂ containing toothpaste on teeth with erosion lesions in the μ SBS of the SBU to enamel and dentin. 120 bovine incisors were randomly divided into 12 groups- (n=10) according to: a) substrate: enamel/dentin; b) substrate condition: sound/eroded (immersion in cola based drink)/eroded-treated (immersion in cola based drink-toothpaste application); c) SBU application strategy: etch-and-rinse (ER)/self-etching (SE). Four composite resin cylinders (0.96mm-diameter/1mm-height) were built on each flat buccal surface. 2 cylinders were tested at each evaluation time. For μ SBS evaluation, the microshear test was performed immediately after 24 hours and after 6 months of storage (distilled water, 37°C). μ SBS-(Mpa) values were analyzed using three-factor ANOVA. In dentin, there was significant cross-interaction among the three factors observed (P=0.023). Eroded dentin reduced μ SBS-(24h) in ER. Treatment with anti-erosive toothpaste also reduced μ SBS values to dentin, regardless of the strategy used. In enamel, there was no significant interaction between the factors. The isolated factors, substrate condition (P=0.026), application strategy (P=0.004) and storage time (P=0.016) were significant. The eroded enamel had no impact on μ SBS, compared to sound enamel. The erosive challenge negatively impacts the μ SBS of the SBU, in dentin, used in the ER strategy, while the treatment with anti-erosive toothpaste compromises the adhesion to dentin in both strategies. Both the erosive challenge and the treatment do not impair enamel adhesion. The aim of the second study was to evaluate the effect of the erosive challenge after adhesive and restorative procedures on enamel and dentin, on the μ SBS stability of the SBU adhesive applied in the two etching strategies. 80 bovine incisors were randomly assigned into 8 groups-(n=10) according to: a) substrate: enamel/dentin; b) SBU application strategy: ER/SE; c) challenge after restoration: erosive challenge in cola drink/storage in artificial saliva. Four composite resin cylinders were built on each flat surface, 2 of which were submitted to the microshear test to determine the μ SBS, immediately after the erosive challenge and storage in artificial saliva for 7 days and 2 after 6 months of storage (distilled water, 37°C). μ SBS-(Mpa) values were analyzed using three-factor ANOVA. The isolated factors, challenge after restoration, application strategy and storage time were statistically significant (P=0.000, P=0.048 and P=0.000, respectively). The μ SBS values in eroded dentin were reduced in ER strategy. The μ SBS stability to dentin was affected in the control group. However, lower immediate μ SBS values were found in the eroded group. In enamel, statistical significance was observed for the application strategy isolated factor (P=0.00), the μ SBS values in ER were higher than in SE group. The erosive challenge after restorative procedures does not compromise the stability of adhesion to enamel and dentin; however, it impairs the immediate μ SBS to the dentin, when the ER strategy is used.

Key words: Bonding, Dental. Erosion, Dental. Strength, Shear.

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1 INTRODUÇÃO

A erosão dental é uma condição multifatorial que envolve a interação de elementos químicos, biológicos e comportamentais. Essa desordem se caracteriza pela perda de estrutura dental patológica e crônica causada por ácidos sem envolvimento bacteriano (LINNETT; SEOW, 2001; TEN CATE; IMFELD, 1996). O desenvolvimento das lesões de erosão dental está diretamente relacionado às propriedades fisiológicas da saliva, às características estruturais dos dentes e aos hábitos dos pacientes, os quais podem contribuir para o desenvolvimento de tal distúrbio (LUSSI et al., 2011).

Os ácidos responsáveis por desencadear as lesões podem ser provenientes de fontes intrínsecas como distúrbios alimentares ou refluxo gástrico (SCHEUTZEL 1996), ou ainda de fontes extrínsecas por meio de uma dieta ácida, ocupações que propiciam o desenvolvimento desse distúrbio, como trabalhadores de fábricas ou laboratórios expostos à vapores ácidos ou aerossóis (ZERO 1996), e até mesmo certos esportes praticados pelos indivíduos, através da exposição direta como a de nadadores à água com baixo pH ou indireta como atletas consumindo com frequência bebidas esportivas (LUSSI; JAEGGI, 2008). O consumo de bebidas e alimentos ácidos é um fator de alta relevância devido às atuais mudanças no estilo de vida das populações. Tanto um estilo de vida saudável, com alto consumo de frutas e sucos, quanto a prática de hábitos não saudáveis, como o consumo de álcool (ROBB; SMITH, 1990), por exemplo, podem aumentar o risco de desenvolver lesões de erosão.

Os fatores comportamentais envolvem, além da frequência, a forma como os ácidos são consumidos e o tempo que permanecem em contato com os tecidos dentais, que podem aumentar o prejuízo aos tecidos (JOHANSON et al., 2004). Parâmetros importantes a serem observados com relação à ingestão de ácidos incluem não apenas o pH da solução, mas também a concentração, o grau de saturação e as concentrações de cálcio e fosfato (BARBOUR; LUSSI; SHELLIS, 2011). Dentre os fatores fisiológicos que podem estar relacionados à erosão, características da saliva, como fluxo salivar, têm importante papel na ocorrência de erosão (JÄRVINEN; RYTÖMAA; HEINONEN, 1991). Vários mecanismos de proteção estão relacionados à saliva durante os desafios erosivos, já que esta apresenta a capacidade diluir os ácidos e promover a neutralização e tamponamento desses agentes erosivos (BUZALAF; HANNAS; KATO, 2012), além da formação da película

adquirida, a qual age como uma barreira de difusão na superfície dos tecidos dentários (HANNING; BALZ, 1999). Além disso, a saliva é supersaturada em relação ao mineral do dente, fornecendo cálcio, fosfato e flúor (BUZALAF; HANNAS; KATO, 2012).

A erosão dos tecidos dentários tem início com uma redução da dureza na superfície do esmalte, que é seguida pela dissolução dos cristais, camada por camada, gerando uma superfície de aparência lisa, polida e, em estágios avançados, a dentina começa a ser exposta, podendo acarretar episódios de hipersensibilidade (LUSSI et al., 2011). O diagnóstico das lesões em estágios iniciais pode ser difícil, dessa forma, muitas vezes evoluem à ponto de necessitar de tratamento restaurador, tanto em esmalte quanto em dentina, a fim de devolver a morfologia perdida visando melhorar estética, função, e/ou prevenir progressão adicional (JAEGGI; GRUNINGER; LUSSI, 2006). Essa desordem chama atenção dos pesquisadores por ser uma condição crescente no que diz respeito a prevalência, que pode afetar tanto a dentição decídua quanto a permanente (JAEGGI; LUSSI, 2014). No entanto, as variações de índices utilizados, amostragens e desenhos de estudo, tornam difícil uma comparação global e a estimativa de valores de prevalência (SCHLUETER; LUKA, 2018).

O crescente número de casos de lesões de erosão dental em adultos e jovens, tem gerado interesse em formas de preveni-las e tratá-las. A ação de diversos produtos odontológicos contendo íons metálicos vem sendo largamente estudada (ASSUNÇÃO et al., 2018; DA SILVA, NAZELLO, DE FREITAS, 2017; JOÃO SOUZA et al., 2017; PINI et al., 2017; RAMOS-OLIVEIRA et al., 2017; SAKAE et al., 2018; ZUMSTEIN et al., 2018), como exemplo os produtos contendo estanho na formulação, que são conhecidos por protegerem os tecidos da desmineralização (RØLLA; ELLINGSEN, 1994). A ação desses produtos se baseia na formação de sais como Sn_2OHPO_4 , $\text{Sn}_3\text{F}_3\text{PO}_4$ e $\text{Ca}(\text{SnF}_3)_2$ que se originam da reação dos íons de estanho com os tecidos dentais (BABCOCK; KING; JORDAN, 1978) formando uma camada resistente à dissolução por ácidos (HOVE et al., 2006). Tanto no esmalte hígido como no erodido, a camada rica em estanho é capaz de ser formada, porém, no primeiro, essa camada fica restrita à superfície, enquanto que no segundo a presença do estanho pode ser observada em uma profundidade de até 20 μm (SCHLUETER et al., 2009), portanto, produtos contendo estanho atuam no esmalte erodido depositando o metal tanto na superfície como incorporando-o nas camadas mais internas (LUSSI; CARVALHO, 2015).

Estudos *in situ* e *in vitro* realizados com soluções e dentifrícios contendo estanho, apresentaram resultados bastante favoráveis com relação à perda de tecido e redução de dureza. (DA SILVA, NAZELLO, DE FREITAS, 2017; JOÃO SOUZA et al., 2017; PINI et al., 2017; SAKAE et al., 2018). Hooper et al. 2014 mostrou, em uma avaliação feita por meio de perfilometria de contato, que um dentifício contendo estanho reduziu 38% da perda de esmalte após 15 dias quando comparado a um dentifício contendo fluoreto de sódio (HOOPER et al., 2014). Schlueter et al., 2013 constatou que um dentifício contendo estanho foi eficaz tanto com um modelo experimental utilizando escovação, quanto apenas com o contato da substância na superfície dental, reduzindo a perda de esmalte em mais de 50% (SCHLUETER; KLIMEK; GANSS, 2013). Porém, é possível observar que a ação dos dentifrícios está relacionada a dificultar a perda de esmalte, protegendo contra erosão inibindo a desmineralização da superfície e não à remineralizá-lo (LUSSI; CARVALHO, 2015). Em estudo realizado em dentina o estanho também foi capaz de reduzir significativamente a perda de superfície em comparação ao grupo controle com água deionizada (ALGARNI, LIPPERT, HARA, 2015).

Nos casos mais avançados, onde o tratamento restaurador torna-se necessário, materiais adesivos são frequentemente utilizados. Os sistemas adesivos disponíveis atualmente são classificados de acordo com a estratégia de aplicação utilizada, sendo divididos em dois grandes grupos: autocondicionantes (self-etch) e com condicionamento ácido prévio (etch-and-rinse) (PASHLEY et al., 2011; VAN MEERBEEK et al., 2003, 2011). Sistemas adesivos autocondicionantes de passo único, que permitem sua aplicação nas duas estratégias, têm sido formulados em busca de reduzir o tempo de aplicação e oferecer versatilidade (DE GOES; SHINOHARA; FREITAS, 2014; HANABUSA et al., 2012). Esses materiais são chamados de adesivos “universais” ou “multi-modo” e permitirão ao profissional optar pelo protocolo mais adequado para cada situação clínica (CARDENAS et al., 2016; HANABUSA et al., 2012; MUÑOZ et al., 2013; WAGNER et al., 2014).

O sistema de condicionamento ácido prévio promove adesão ao esmalte por meio da dissolução seletiva dos cristais de hidroxiapatita, produzindo microporosidades profundas que serão ocupadas pelo adesivo por capilaridade, possibilitando a formação de tags (VAN MEERBEEK et al., 2003), já em dentina gera a desmineralização de alguns micrômetros, expondo fibras colágenas desprovidas de hidroxiapatita que serão infiltradas pelos monômeros (LOGUERCIO et al., 2005). De

outra forma, os adesivos auto-condicionantes não requerem um passo exclusivo para o condicionamento, pois possuem em sua composição monômeros acídicos que condicionam a superfície e infiltram no espaço simultaneamente (VAN MEERBEEK et al., 2011). Tais materiais não removem a smear layer da superfície, mas a incorporam à camada hibridizada (VAN MEERBEEK et al., 2011).

Apesar de sua relevância, um número limitado de estudos avaliou a adesão aos substratos erodidos. Tanto em esmalte quanto em dentina os resultados de estudos que avaliaram resistência de união são controversos (CASAS_APAYCO et al., 2014; CRUZ et al., 2012; FLURY et al., 2013; FRATTES et al., 2017; LENZI et al., 2013; ZIMMERLI et al., 2012; WANG et al., 2014;). Sendo incerta a durabilidade das restaurações nesses substratos. (ZIMMERLI et al., 2012)

Além disso, levando em consideração que o uso de substâncias contendo estanho com a finalidade de prevenir as lesões de erosão pretende formar uma camada resistente à ação de ácidos (HOVE et al., 2006), e que a adesão aos substratos dentários é baseada na desmineralização da superfície por meio de ácidos, independentemente da estratégia adesiva utilizada, a resistência de união de sistemas adesivos à substratos submetidos ao uso dessas substâncias ainda é pouco conhecida.

Sabe-se que a longevidade das restaurações é necessária para indicar o sucesso de tais procedimentos (JAECCI; GRUNINGER; LUSSI, 2006) e que a integridade dos materiais restauradores pode ser afetada por novos desafios erosivos (FRANCISCONI et al., 2008). Tendo em conta que os tecidos adjacentes e os materiais restauradores, bem como sua interface, continuam expostos ao meio bucal e que os distúrbios erosivos estão diretamente relacionados à hábitos e comportamentos, fatores de difícil modificação, é provável que tais desafios continuem a ocorrer após os procedimentos restauradores. Esse contexto torna relevante a avaliação o efeito dos agentes erosivos sobre a interface adesiva, no que diz respeito à resistência de união.

Considerando todo o cenário acima exposto, a presente tese de doutorado traz dois artigos:

O primeiro, intitulado “***Does the use of anti-erosive toothpaste influence the bonding effectiveness of a universal adhesive system?***” avaliou, por meio de um estudo *in vitro*, o efeito de um dentifrício contendo Cloreto de Estanho - SnCl₂ na resistência de união de um sistema adesivo Universal à substratos erodidos.

O segundo artigo, também realizado in vitro, intitulado “***Erosive challenge impairs the dentin bond stability of a universal adhesive***” analisou a estabilidade da resistência de união de um adesivo universal, ao esmalte e à dentina submetidos ao desafio erosivo após restaurados.

2 ARTIGO 1 - DOES THE USE OF ANTI-EROSIVE TOOTHPASTE INFLUENCE THE BONDING EFFECTIVENESS OF A UNIVERSAL ADHESIVE SYSTEM?

Este artigo será submetido ao periódico *The Journal of Adhesive Dentistry*, ISSN: 1461-5185, Fator de impacto = 1.311; Qualis A2. As normas para publicação estão descritas no Anexo A.

**DOES THE USE OF ANTI-EROSIVE TOOTHPASTE INFLUENCE THE BONDING
EFFECTIVENESS OF A UNIVERSAL ADHESIVE SYSTEM?**

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DOES THE USE OF ANTI-EROSIVE TOOTHPASTE INFLUENCE THE BONDING EFFECTIVENESS OF A UNIVERSAL ADHESIVE SYSTEM?

Abstract

Purpose: To evaluate the effect of an anti-erosive toothpaste containing SnCl₂ on the bond strength of Scotchbond Universal Adhesive (SBU – 3M Oral Care), used in etch-and-rinse (ER) and self-etch (SE) strategies to eroded enamel and dentin. *Materials and methods:* Buccal flat enamel and dentin surfaces of 120 sound bovine incisors were randomly assigned to 6 groups (n=10) per substrate, according to substrate condition (sound - SND, eroded – immersion into cola drink - ERO and eroded + treated – SnCl₂ containing toothpaste - TRE), and etching modes (SE or ER). The adhesive system was applied according to the manufacturer's recommendations, and four starch tubes were positioned on each surface and light-cured. The tubes were filled with resin composite. After 24h of distilled water storage (37°C), the tubes were removed, and two specimens were submitted immediately (24h) and two after six months (6mo) to microshear bond strength test (μSBS). Bond strength values (MPa) were analyzed by three-way ANOVA. *Results:* Eroded dentin significantly reduced bond strength values (24h) in the ER strategy. The treatment with SnCl₂ containing toothpaste also reduced dentin's bond strength, regardless the etching strategy. Eroded enamel did not impair the bond strength compared to the sound while treated enamel produced higher bond strength values than sound and similar values compared to eroded. *Conclusion:* Eroded dentin negatively impacts the bond strength of SBU used in the ER strategy, whereas treated dentin compromised the adhesion in both strategies. Erosion or treatment with anti-erosive toothpaste do not impair the bonding to enamel.

Keywords: Dental Bonding, Shear Strength, Tooth Erosion

Introduction

Dental erosion is a common and crescent multifactorial condition^{23,17}, defined as dental mineral loss caused by acids of non-bacterial origin^{5,21}. Lesions development occurs through a chemical dissolution process, and it is difficult to diagnose in incipient stages^{22,23}. The dissolution can be by both intrinsic or extrinsic acid sources, and the impact varies according to the solutions involved, pH, and exposure time¹⁹. The extent of mineral loss depends, further on the chemical aspect, on biological and behavioral factors²³. Thus, saliva plays an important role in this process,³ and the frequency and the manner these acids are consumed¹⁹. Exposing the dental tissues to these erosive episodes leads to a tooth surface softening and, as it advances into the enamel, can cause mineral dissolution to the point where there is complete loss of the enamel layer.²⁰

Despite the variations in the erosion indices used³⁸, studies show that dental erosion increasingly affects adults and children¹⁷. This increase in the prevalence has led to the interest in finding ways to prevent and treat the disorder. Since the isolated action of fluoride on erosion prevention is limited, the combination with other protective agents shows more promise. Products that contain polyvalent metal ions, such as those that contain tin, and some polymers in the composition are examples²⁴. In an attempt to reduce the erosive process progression, the action of dental products containing tin ions has been widely explored, and it has shown promising results in protecting dental hard tissues from demineralization^{2,7,18,30,32,35}. The action of these ions is based on the deposition and incorporation of tin in the superficial layers of the tooth, making it less prone to demineralization²⁴. Although the use of oral hygiene products as a non-invasive strategy to inhibit mineral loss is an option, the advancement of the erosive process can lead to the formation of lesions, from yellowish color to changes in shape, added to hypersensitivity in advanced stages, leading to restorative approach need¹⁶.

In cases where restorative treatment becomes necessary, adhesive materials are frequently used. Restoration of non-cariious cervical lesions with adhesive materials can improve structural integrity and biomechanical function around the lesion¹⁶. Thus, it is essential to know the performance of adhesives on altered substrates, either by erosion or by the action of substances used in the lesion's

treatment. The universal or multi-mode adhesives are the latest development that aims to simplify the procedures promoting stable adhesion to the dental substrates, being able to be used in both application strategies^{15,28,41}. Regardless of the strategy, adhesive systems' performance depends on the interaction with the demineralized substrate^{27,29}. Therefore, considering that the use of tin-containing materials is intended to form a barrier that prevents demineralization, the bond strength of adhesives on a surface previously treated with these materials becomes unclear.

Thus, this investigation aimed to evaluate the influence of anti-erosive toothpaste containing SnCl₂ on the bond strength of Scotchbond Universal Adhesive, used in etch-and-rinse and self-etch strategies to eroded enamel and dentin. The null hypothesis was that the eroded substrate or the use of tin-containing toothpaste has no influence on the bond strength values of a universal adhesive to enamel and dentin, regardless of the etching approach and storage time.

Materials and methods

Study design

Enamel or dentin surfaces of bovine teeth were randomly (RANDOM.ORG) allocated into 6 groups (n=10) per substrate, according to the substrate condition – sound, eroded or eroded+treated, and the etching modes – self-etch or etch-and-rinse.

Tooth selection and preparation

One hundred and twenty freshly extracted bovine incisors were selected, cleaned, disinfected in 0,5% aqueous chloramine, and stored in distilled water until use. The root portion was removed using a diamond saw in a low-speed handpiece. Crowns were partially embedded in self-curing acrylic resin inside PVC rings (JET clássico, São Paulo, SP, Brazil), leaving the buccal surfaces exposed. The exposed surfaces were ground with 180-grit SiC paper under running water to create flat enamel or dentin surfaces. Then, the enamel and dentin surfaces were ground for 60-s with 600-grit SiC paper to standardize surface smoothness and smear layer, respectively.

Artificially induction of eroded enamel and dentin (ERO)

Eighty specimens were subject to erosive challenge through a cola drink immersion (Coca-Cola®, [pH—2.6, phosphate—5.43mM Pi, Calcium—0.84 mM Ca²⁺, Fluoride—0.13 ppm F, titratable acid—40.0 mmol/l OH⁻ to pH 5.5 and 83.6 mmol/l OH⁻ to pH 7.0], Spal, Porto Real, RJ, Brazil), three times a day (5 minutes under agitation) for seven days and kept in artificial saliva at room temperature (1.5 mmol/l—1 Ca[NO₃]₂·4H₂O, 0.9 mmol/l—1 NaH₂PO₄·2 H₂O, 150 mmol/l—1 KCl, 0.1 mol/l—1 Tris buffer, 0.03 ppm F, pH 7.0) in the remaining time¹¹. Between the cycles, specimens were washed with deionized water, dried with absorbent paper, and the storage solution was renewed.

SnCl₂ tooth paste application (TRE)

The ELMEX erosion protection toothpaste (Colgate-Palmolive, Switzerland) was used (composition described in Table 1). The toothpaste was mixed with a mineral salt solution (1 part toothpaste and 3 parts mineral salt solution by weight). The solution was supersaturated in relation to hydroxyapatite (4,08 mmol de H₃PO₄, 11,90 mmol NaHCO₃, 20,10 mmol KCl e 1,98 mmol CaCl₂)¹⁴. Previously eroded specimens were immersed daily after the first and the last cycle for 2 min in the solution/toothpaste³⁷.

Bonding and restorative procedures

Scotchbond Universal Adhesive (3M Oral Care, St. Paul, MN, USA) (SBU) was used in self-etch (SE) and etch-and-rinse (ER) strategies. The adhesive was applied by a single operator following the manufacturer's recommendations on four areas of each enamel and dentin surface. Starch tubes (0,96mm internal diameter x 1mm height) were positioned and light-cured (Radii-cal, SDI, Bayswater, Victoria, Australia). The tubes were filled with resin composite (Z250, 3M Oral Care, St. Paul, MN, USA, shade A3) and light-cured for 20 s. Four cylinders of composite resin were obtained for each specimen. The SBU composition and manufacturer's instructions are described in Table 2.

Microshear bond strength test (μ SBS)

After 24h storage (distilled water, 37° C), starch tubes were removed using air/water spray. Two random cylinders of each specimen were submitted immediately (24h) to microshear bond strength test in a universal machine (Emic DL 1000, Equipment and Systems; São José dos Pinhais, PR, Brazil) using a stainless-steel wire loop (0,2mm in diameter) placed as close as possible to the resin-enamel/dentin interface. The shear load was applied at a crosshead speed of 1mm/min until failure. The remaining two cylinders were evaluated after additional six months of water storage (6mo). For cylinders that failed before the test, a value of 0 MPa was assigned, and they considered in the statistical analysis. All tests were performed by a single blinded operator.

Failure Mode

The fractured specimens were examined under a stereomicroscope (Discovery V20, Carl Zeiss, Gottingen, Germany) at 40X magnification to determine the failure mode and categorized as mixed/adhesive, cohesive in enamel/dentin, or cohesive in resin composite.

Statistical Analysis

Statistical analysis was performed separately for enamel and dentin. The tooth was the experimental unit of the study. The normal distribution of data was tested with the Anderson-Darling test, and enamel and dentin data were submitted to a three-way Analysis of Variance (ANOVA), considering the substrate condition, etching strategy, and time as the main factors.

Results

Dentin

Three-way ANOVA showed significant cross-interaction ($P=0.023$) on dentin among the three factors (substrate condition x etching strategy x storage time). Table

3 presents the μ SBS values (means and standard deviation), pre-testing failures, and contrasts found in the interaction.

Eroded dentin significantly reduced bond strength values in immediate analysis (24h) when phosphoric acid was used (ER strategy), and similar results were observed after 6mo storage. On the other hand, with the SE strategy, bond strength values were similar between sound and eroded dentin, with a significant reduction after 6 months in eroded group.

The treatment with SnCl_2 containing toothpaste also produced lower bond strength values than sound dentin, regardless of the etching strategy or storage time. Compared to eroded dentin, eroded+treated substrate produces similar bonding values in the ER strategy at 24h but lower after water storage. On the opposite, when SBU was used in the SE strategy, lower values were only found when specimens were tested immediately (24h). No pre-testing failures occurred in the sound substrate, whereas treated dentin produced apparently more ptf's than eroded, especially on 6mo analysis for both strategies. All failures observed were classified as adhesive.

Enamel

For enamel, three-way ANOVA showed significance for the three main factors - substrate condition ($P=0.026$), etching strategy ($P=0.004$), and storage time ($P=0.016$). No interaction between the main factors nor the triple interaction was statistically significant. Table 4 presents the μ SBS values and standard deviation for experimental groups, and Table 5 presents the means and standard deviations for the main factors. Eroded enamel did not impair the bond strength compared to the sound substrate. Treated substrate, however, produced higher bond strength values than sound enamel and similar values compared to eroded. The etch-and-rinse strategy and the storage time also resulted in higher bonding values.

No pre-testing failures were observed in enamel analysis, and all failures were adhesive.

Discussion

The increase in dental erosion prevalence requires concern about the treatment of the lesions, whether to stop its progression or perform a restorative treatment. Adhesive restorations are often necessary when the dental structure was severely

lossed¹⁶, given the difficulty of controlling etiological factors. However, both the treatment and the substrate itself altered by the erosive process can compromise the adhesion of adhesive systems to enamel and dentin. In the present study, the effect of eroded substrate and the treatment with tin-containing toothpaste was distinct for enamel and dentin. Thus, the null hypothesis that these factors had no influence on the bond strength values can be partially rejected.

Different results were found for enamel and dentin, which is expected due to the vast histological and morphological differences of these two substrates. As previously described³⁴, in sound dentin, the prior acid etching did not improve the bond strength of the universal adhesive. In addition, it did not affect the bond strength stability after six months of water storage. The literature suggests that in dentin, artificial erosion challenges lead to surface demineralization, removing the organic intertubular dentin and dentinal plugs, increasing tubule diameter, and exposing collagen matrix³¹. The bonding to eroded substrate seems to be impaired compared to sound dentin^{6,10,39}, and in this study, it was observed when a prior acid-etching technique was used. In the self-etching strategy, the universal adhesive produced similar immediate values in sound and eroded dentin, which was also noticed in previous research that used the same adhesive system¹². However, bond degradation, evidenced by lower bond strength values, was seen for eroded dentin, regardless of the strategy.

The etching strategy was not significant when SBU was applied to treated dentin. When dealing with dentin previously treated with tin-containing fluoridated substances, little is known about the interaction of the product with this substrate. Gans et al¹³ showed that tin is deposited on the dentin surface, especially in the presence of acid challenges, and is incorporated into the tissue, demineralized or not. A study that evaluated the microtensile bond strength of a self-etching adhesive to dentin treated with tin-containing fluoridated mouth rinse found no difference in bond strength values between sound and treated dentin without removing the demineralized organic matrix⁹. It is worth noting that, in addition to different tests, that study used a two-step self-etching adhesive without active application, which could have altered the structure and organic matrix of the eroded dentin, which may have been advantageous for the results.

Eroded enamel did not decrease the bond strength compared to sound, the same was observed previously using *in situ* conditions, in which, eroded enamel exhibited a rougher surface⁴². Despite a rougher surface increases the

micromechanical interlocking, an essential aspect in the adhesion mechanism²⁵, the fact that there is no difference between the substrates may suggest an interference of saliva in this process⁴².

Although the mechanism of action of tin-containing products in eroded enamel occurs through tin deposition on the tooth surface and the eventual incorporation into layers close to the enamel surface, making it less susceptible to dissolution²⁴, statistically high bond strength values were found in this substrate compared to sound enamel. A previous study evaluated the microtensile bond strength of different adhesive systems to previously treated enamel with a concentrated acidic SnCl₂ solution, which increased the bond strength of a self-etching adhesive to the enamel.³⁶

According to the literature, using the acid etching strategy before applying multi-mode adhesives on sound enamel improves bond strength values^{1,4,8,15,26,34}. The same was observed, as overall, the etch-and-rinse strategy resulted in higher bonding values, regardless of substrate condition and storage time.

An erosive challenge using a cola drink was used to simulate the dental erosion because this beverage has a high erosive potential³³ due to low pH and low fluoride and calcium concentration¹¹. It is worth noting that although only one universal adhesive was evaluated in this study, its performance in different forms of application on both substrates was observed. The use of bovine teeth is well accepted, especially for tests that evaluate adhesion to dental tissues, as they produce resembling bond strength results to human teeth⁴⁰. Furthermore, the use of bovine incisors has the benefit of being easy to obtain in large quantities and the ease of standardizing the tooth age. Therefore, studies that assess the bond strength stability for longer storage times should be encouraged.

Conclusion

The effect of anti-erosive toothpaste containing SnCl₂ on the bond strength of Scotchbond Universal Adhesive was different in enamel and dentin. Erosion reduces the bond strength of Scotchbond Universal Adhesive to dentin, only in the etch-and-rinse strategy. The anti-erosive toothpaste treatment impairs the dentin bond strength,

regardless of the etching strategy. The adhesion to enamel is not compromised by erosion or treatment.

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Clinical significance

When dealing with patients suffering dental erosion or using tin-containing fluoridated toothpaste, we must consider the performance of the adhesive system applied in different etching modes to decide for each type of substrate.

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Tables

Table 1: ELMEX erosion protection toothpaste composition

Product	Composition	Active agents
Elmex Erosion Protection Toothpaste	Aqua, glycerin, sorbitol, hydrated silica, hydroxyethylcellulose, aroma, cocamidopropyl betaine, sodium gluconate, alumina, sodium saccharin, potassium hydroxide, hydrochloric acid	1400 ppm F- (AmF/NaF) 3500 ppm Sn ²⁺ (SnCl ₂) 0.5% chitosan

Table 2: Scotchbond Universal composition and application mode

Mode	Self-etching	Etch-and-rinse
Application mode*	Apply the adhesive for 20 s with vigorous agitation Gently air thin for 5 s Light cure for 10 s	Apply etchant for 15 s Rinse for 10 s Air dry to remove excess water Apply the adhesive as for the self-etching mode
Components (pH 2,7)	Etchant: 34% phosphoric acid, water, synthetic amorphous silica, polyethylene glycol, aluminium oxide Adhesive: MDP phosphate monomer, dimethacrylate resins, HEMA, methacrylat emodified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane	

*According to the manufactures' instructions.

Table 3: Dentin μ SBS mean values (Mpa), standard deviation and pre-testing failure (ptf) for experimental groups

Etching strategy	ER				SE			
	24h		6mo		24h		6mo	
Storage time	Mean (sd)	ptf	Mean (sd)	ptf	Mean (sd)	ptf	Mean (sd)	ptf
Sound	18.1 (3.8) ^A	0	14.5 (4.3) ^{A, B}	0	15.1 (3.9) ^{A, B}	0	13.8 (3.0) ^{A, B, C}	0
Eroded	11.9 (3.0) ^{B, C}	0	10 (5.7) ^{B, C, D}	3	15.1 (3.3) ^{A, B}	0	8.2 (3.6) ^{C, D, E}	5
Treated	9.8 (4.1) ^{B, C, D}	3	2.5 (2.1) ^E	10	8.2 (4.7) ^{C, D, E}	5	5.3 (4.4) ^{D, E}	10

*different letters indicate significance

Table 4: Enamel μ SBS mean values in MPa and standard deviation for experimental groups

Etching strategy	ER		SE	
	24h	6mo	24h	6mo
Sound	21.8 (4.8)	20.8 (3.9)	15.9 (3.8)	17.6 (3.4)
Eroded	19.4 (3.2)	21.5 (4.5)	18.9 (3.6)	21.3 (3.0)
Treated	20.8 (3.3)	23.9 (4.4)	19.5 (3.4)	21.8 (6.3)

Table 5: Enamel μ SBS mean values in Mpa and standard deviation according to the main factors*

Factor	Mean (SD)
Substrate condition	
Sound	19.0 (4.5) ^B
Eroded	20.3 (3.7) ^{A, B}
Treated	21.5 (4.6) ^A
Etching strategy	
ER	21.4 (4.1) ^A
SE	19.2 (4.4) ^B
Storage time	
24 h	19.4 (3.9) ^B
6 mo	21.1 (4.6) ^A

*different letters indicate significance

3 ARTIGO 2 - EROSIVE CHALLENGE IMPAIRS THE DENTIN BOND STABILITY OF A UNIVERSAL ADHESIVE

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EROSIVE CHALLENGE IMPAIRS THE DENTIN BOND STABILITY OF A UNIVERSAL ADHESIVE

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EROSIVE CHALLENGE IMPAIRS THE DENTIN BOND STABILITY OF A UNIVERSAL ADHESIVE

Abstract:

Purpose: To evaluate the effect of erosive challenge on the bond stability of Scotchbond Universal (SBU - 3M Oral Care) applied in etch-and-rinse (ER) or self-etch (SE) strategies to enamel and dentin. *Materials and methods:* Buccal flat enamel and dentin surfaces of eighty sound bovine incisors were randomly assigned to 8 groups (n=10) according to the substrate (dentin or enamel), etching strategy, and challenge after bonding (erosive challenge by immersion in cola drink [ERO] or artificial saliva [control - CTR]). Four starch tubes were positioned on each buccal surface, light-cured, and completely filled with resin composite. After 7 days of erosive challenge (5 minutes immersion, 3 times/day) or artificial saliva storage, half of the specimens were subjected to a microshear bond strength test (μ SBS). The other two specimens were tested after six months (6mo). Bond strength values (MPa) were analyzed separately for enamel and dentin by three-way ANOVA, considering etching strategy, challenge after bonding, and time as factors. *Results:* ERO significantly reduced dentin bond strength for ER strategy. Dentin bonding stability was affected only in the control group. However, lower immediate values were found for the ERO group. In enamel, only ER strategy influenced the bond strength values, presenting higher values than SE. No other comparison was statistically significant. *Conclusion:* The erosive challenge after bonding procedures does not compromise the bond stability of SBU to enamel and dentin; however impairs the immediate bond strength to dentin when the ER strategy was used.

Keywords: Shear strength, dental bonding, tooth erosion.

Introduction

In recent years, dental erosion has attracted attention due to its increasing prevalence¹³. Some factors such as changes in the lifestyle, increased consumption of soft drinks and other diet-related behaviors, and gastroesophageal disorders are responsible for this increase in cases^{5,6,15,16}. Tooth erosion is characterized by a progressive loss of dental hard tissue caused by acids of extrinsic or intrinsic origin³² without involving microorganisms^{3,14}. As a multifactorial disease, it involves, in addition to chemical factors, biological and behavioral components¹⁶.

The initial stages, which are difficult to diagnose, are restricted to the enamel. Given the difficulty of controlling the etiological factors, dentin exposure may occur as the lesion progresses, leading to an unacceptable appearance or even dentin hypersensitivity^{1,12}. When restorative treatment becomes necessary, adhesive materials are frequently used to restore esthetics, function and prevent the progression of tooth structure loss²³.

The use of universal adhesive systems, also called multi-mode adhesives, has become common among professionals due to their versatility. Such adhesives can be applied with prior acid etching or in the self-etching mode, and it is up to the professional to decide the most appropriate form of use in different clinical situations^{11,20,31}. Although the lesion's restoration is intended to reduce the progression of the erosion process¹³, the adjacent tissues and the restorative materials are still exposed to the oral environment. Considering that the development of dental erosion involves biological and behavioral factors and facing the difficulty of changing habits, the oral environment is likely to continue suffering new erosive challenges.

The longevity of adhesive restorations is essential to indicate treatment success¹³. Some factors, including the presence of acid challenges, can affect the restoration's durability¹². Degradation at the adhesive interface can affect the performance of restorations⁶. Given this scenario, it is important to know the behavior of adhesive restorations with a universal adhesive applied in both application modes under the continuity of erosive challenges.

Thus, this study aimed to evaluate the effect of erosive challenge on the bond stability of a universal adhesive system used in two etching strategies to enamel and dentin. The null hypothesis was that the erosive challenge after bonding procedures

has no influence on the bond strength values of Scotchbond Universal Adhesive to enamel and dentin, regardless the etching strategies and storage time.

Materials and methods

Study design

Enamel and dentin surfaces were, separately, were randomly allocated into 2 groups (n=10), according to the etching strategy (self-etch or etch-and-rinse), and challenge after bonding procedures (erosive challenge or artificial saliva).

Tooth preparation

Eighty freshly extracted bovine incisors were selected, cleaned, disinfected in 0,5% aqueous chloramine, and stored in distilled water until use. The root portion was removed using a diamond saw in a low-speed handpiece. Crowns were partially embedded in self-curing acrylic resin inside PVC rings (JET clássico, São Paulo, SP, Brazil), leaving the buccal surfaces exposed. The exposed surfaces were ground with 180-grit SiC paper under running water to create flat surfaces. Then, the enamel and dentin surfaces were ground for 60s with 600-grit SiC paper to standardize of surface smoothness and smear layer, respectively.

Bonding and restorative procedures

Scotchbond Universal Adhesive (SBU) was used in self-etch (SE) and etch-and-rinse (ER) strategies. A single operator applied the adhesive following the manufacturer's recommendations on four areas of each enamel and dentin surface. Starch tubes (0.96mm internal diameter x 1mm height) were positioned and light-cured (Radii-cal, SDI, Bayswater, Victoria, Australia). The tubes were completely filled with resin composite (Z250, 3M ESPE, St. Paul, MN, USA, shade A3) and light-cured for 20s. Four cylinders of composite resin were obtained for each specimen. The adhesive system and composite resin's composition and manufacturer instructions are described in Table1.

Erosive challenge

Forty specimens were subject to erosive challenge through a cola drink immersion (Coca-Cola®, [pH—2.6, phosphate—5.43mM Pi, Calcium—0.84 mM Ca²⁺, Fluoride—0.13 ppm F, titratable acid—40.0 mmol/l OH⁻ to pH 5.5 and 83.6 mmol/l OH⁻ to pH 7.0], Spal, Porto Real, RJ, Brazil), three times a day (5 minutes under agitation) for seven days. In the remaining time, specimens were kept in artificial saliva at room temperature (1.5 mmol/l—1 Ca[NO₃]₂.4H₂O, 0.9 mmol/l—1 NaH₂PO₄.2 H₂O, 150 mmol/l—1 KCl, 0.1 mol/l—1 Tris buffer, 0.03 ppm F, pH 7.0)⁹. Between the cycles, specimens were washed with deionized water, dried with absorbent paper, and storage solution was replaced.

Control group

Forty specimens remained immersed in artificial saliva for 7 days, equivalent to the erosive challenge, after the restorative procedure. The storage solution was changed daily.

Microshear bond strength test (μSBS)

The starch tubes were removed with an air/water spray. Next, the specimens were inspected under a stereoscope at 10x magnification (Discovery V20, Carl Zeiss, Gottingen, Germany) to ensure the absence of bubbles in the adhesive interface. After the challenge after bonding, two cylinders of each specimen were subjected immediately to the microshear test, using a stainless-steel wire loop (0,2mm in diameter) next to the adhesive interface, in a universal machine (Emic DL 1000, Equipment and Systems; São José dos Pinhais, PR, Brazil). The shear load was applied at a crosshead speed of 1mm/min until failure. The remaining specimens were evaluated after additional six months of storage in distilled water at 37 ° C. The tests were performed by a single blinded operator.

Failure Mode

The fractured specimens were examined under a stereomicroscope at 40X magnification to determine the failure mode and categorized as mixed/adhesive, cohesive in enamel/dentin, or cohesive in resin composite

Statistical Analysis

The experimental unit of the study was the tooth. Statistical analysis was performed separately for enamel and dentin. The Anderson-Darling test confirmed normal distribution. Data were submitted to three-way ANOVA, considering challenge after bonding, etching strategy and water storage time as the main factors.

Results

Dentin

Three-way ANOVA showed significance for the three main factors: challenge after bonding, etching strategy, and storage time; as the erosive challenge, SE strategy, and 6 months of water storage impair the bond strength values ($P=0,000$, 0.048 and, 0.000 respectively). The following double interactions were also statistically significant: erosive challenge x etching strategy and erosive challenge x storage time ($P=0.000$ and 0.015 , respectively). Table 2 presents the μ SBS values, standard deviation, and pre-testing failures for experimental groups, and Tables 3 and 4 present the means and standard deviations for the statistically significant interactions. The erosive challenge significantly reduced the dentin bond strength when ER strategy was used, as ER strategy produced higher bonding values than SE strategy in the control group (Table 3).

Considering bond degradation after water storage time, reduced bond strength values were only found for the control group after 6 months; however, it is noticeable that the values for the erosive challenge group were lower already at the immediate evaluation time (Table 4). Most of the pre-testing failures (82%) were observed in the erosive challenge groups, as described in Table 2. All failures observed were classified as adhesive.

Enamel

Table 5 presents the μ SBS values and standard deviation for experimental groups. Three-way ANOVA only pointed out a statistical significance for the main factor etching strategy ($P=0.00$). Overall, phosphoric acid (ER strategy) produced significantly higher bonding values (Table 6) regardless of the challenge after bonding or water storage time. No pre-testing failures were observed, and all failures were adhesive.

Discussion

Several factors can impair the longevity of adhesive restorations, including those related to the patient, like caries and occlusal stress risk and socioeconomic factors⁸. Thus, the chemical process involved in dental erosion can also impact the durability of adhesive restorations, as the erosion challenge process can continue after restorative therapy, compromising the behavior of the adhesive interface.

In the present study, the overall effect of the erosive challenge after bonding jeopardized the bond strength values to dentin; likewise, the self-etch strategy also produced lower bonding to dentin. Thus, considering the interaction between the challenge after bonding and etching strategy, it was possible to note that the higher values were found for the adhesive applied in the etch-and-rinse strategy in the control group (stored in artificial saliva for 7 days). No differences were found between etching strategies when specimens were subjected to the erosive challenge after bonding; that is, considering the etching strategies, the etch-and-rinse mode seems to have been more sensible to chemical challenge.

The subsequent erosive challenge to restorative procedures negatively influenced the bond strength of the SBU to dentin, both immediately and after six months of water storage. This impairment on the bond strength values agrees with a previous study,²⁹ and can be attributed to a dentin demineralization as a consequence of the erosive process, especially in the regions close to the adhesive interface, modifying not only the substrate,^{26,33} but also the adhesive interface itself. The demineralization of the contiguous dentin to the bonding interface can create a zone with exposed collagen fibrils²⁹ and further weakening the dentin-adhesive interface.

On the other hand, the erosive challenge does not jeopardize the bond strength to enamel, which may be related to the fact that enamel is more mineralized, has lower organic content than dentin, and also the stability of the adhesive interface involving enamel is greater²⁹. Furthermore, higher bond strength values to enamel were achieved using the universal adhesive in an etch-and-rinse strategy, as already shown in previous studies reinforcing the use of phosphoric acid before applying universal adhesives to enamel^{2,4,7,10,11,18,25}. In addition, prior phosphoric acid application to enamel increases the surface area and the surface energy, improving the micromechanical adhesion³⁰.

Also, for the enamel substrate, this study showed that six months of storage in water did not influence the bond strength of the SBU, regardless of the strategy used. Enamel bonding stability can be explained by the fact that bonding to sound enamel does not degrade so quickly,¹⁹ considering the water storage time used in the present study. Furthermore, the exposure of enamel interfaces to erosive acid challenge also had no influence on bond strength, which may be related to the simplicity and predictability of adhesion to this substrate^{21,22}.

The erosive challenge used in this research was chosen because cola drink has a high erosive potential²⁴ due to its low pH and low calcium and fluoride concentrations⁹. Although in vitro studies cannot simulate what happens in the oral environment, mainly due to the important participation of saliva and buffering capacity in the process, the model used aims to simulate regular ingestion of soft drinks considered a risk for tooth erosion development³⁴.

It is important to note that the use of bovine teeth in this study, in addition to having the benefit of being easily collected in large quantities and at standardized ages, is well accepted for studies of adhesion to dental tissues, as they achieve bond strength values similar to human's teeth²⁷. However, it should be noted that there are some possible limitations in this study, mainly related to the evaluation of a single universal adhesive, albeit in different application strategies, hindering the extrapolation of the results to other systems. The second limitation concerns the short storage time of the specimens. Even so, dentin bonding degradation was possible to be observed.

From the results of this study, we partially rejected the null hypothesis as erosive challenge produced different outcomes in dentin, depending on the adhesive application strategy and storage period, while enamel was not influenced by the erosive challenge.

Conclusion

The erosive challenge after the bonding procedures compromised dentin bond strength of SBU in etch-and-rinse strategy; enamel bond strength was not impaired.

Clinical significance

Adhesive restorations with dentin margins are more prone to adhesive interface degradation during erosive challenges.

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Tables

Table 1: Scotchbond Universal composition and application mode

Mode	Self-etching	Etch-and-rinse
Application mode*	Apply the adhesive for 20 s with vigorous agitation Gently air thin for 5 s Light cure for 10 s	Apply etchant for 15 s Rinse for 10 s Air dry to remove excess water Apply the adhesive as for the self-etching mode
Components (pH 2,7)	Etchant: 34% phosphoric acid, water, synthetic amorphous silica, polyethylene glycol, aluminium oxide Adhesive: MDP phosphate monomer, dimethacrylate resins, HEMA, methacrylat emodified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane	

*According to the manufactures' instructions.

Table 2: Dentin μ SBS mean values (MPa), standard deviation and pre-testing failure (ptf) for experimental groups

Etching Strategy	ER				SE			
	Immediate		6mo		Immediate		6mo	
Storage time	Mean(SD)	ptf	Mean(SD)	ptf	Mean(SD)	ptf	Mean(SD)	ptf
Control	15.0 (2.2)	0	9.1 (2.9)	0	9.6 (2.1)	0	7.3 (3.6)	5
Erosive challenge	6.4 (2.1)	6	5.4 (1.5)	7	8.0 (1.9)	4	6.5 (3.2)	7

Table 3: Dentin μ SBS mean values in MPa, standart deviation and contrasts for the interaction factors challenge after bonding x etching strategy*

	ER	SE
Control	12.1 (3.9) A	8.5 (3.1) B
Erosive challenge	5.9 (1.9) C	7.2 (2.6) B, C

*Means that do not share a letter are significantly different

Table 4: Dentin μ SBS mean values in MPa and standard deviation and contrasts for the interaction factors challenge after bonding x water storage time*

	Immediate	6mo
Control	12.3 (3.5) A	8.2 (3.3) B
Erosive challenge	7.2 (2.1) B, C	5.9 (2.5) C

*Means that do not share a letter are significantly different

Table 5: Enamel μ SBS mean values in MPa and standard deviation for experimental groups

Etching Strategy	ER		SE	
Storage time	Immediate	6mo	Immediate	6mo
Control	19.0 (2.8)	17.7 (4.3)	12.6 (2.6)	12.7 (3.4)
Erosive challenge	18.8 (3.2)	19.0 (3.3)	14.1 (2.9)	16.0 (2.9)

Table 6: Enamel μ SBS mean values in MPa and standard deviation and contrasts for factor etching strategy *

Etching Strategy	ER	SE
	18.6 (3.4) ^A	13.8 (3.2) ^B

*Means that do not share a letter are significantly different

4 DISCUSSÃO

Os estudos previamente apresentados avaliaram o desempenho de um adesivo Universal aplicado em diferentes estratégias de condicionamento seguindo as recomendações do fabricante. A categoria de adesivos Universais ou multi-modo se destaca pela versatilidade, podendo ser utilizado com condicionamento ácido prévio ou de forma autocondicionante, permitindo ao profissional optar pelo protocolo mais adequado para cada situação clínica (CARDENAS et al., 2016; DE GOES; SHINOHARA; FREITAS, 2014; HANABUSA et al., 2012; MUÑOZ et al., 2013; WAGNER et al., 2014).

Ambos estudos trouxeram a investigação do comportamento desse material, quando utilizado em substratos alterados pelo desafio erosivo, pois, é necessário ter em conta que os profissionais se deparam diariamente com substratos em condições diferentes das quais os adesivos foram originalmente desenvolvidos (TEDESCO et al., 2014). Porém, considerando o grande aumento na prevalência de distúrbios erosivos na população em geral (JAEGGI; LUSI, 2014), a necessidade de conhecer o comportamento desses materiais perante o desafio ácido se torna relevante. Estudos prévios avaliaram o desempenho de sistemas adesivos aplicados sobre esse substrato (CASAS_APAYCO et al., 2014; CRUZ et al., 2012; FLURY et al., 2013; FRATTES et al., 2017; LENZI et al., 2013; WANG et al., 2014; ZIMMERLI et al., 2012) mas o efeito da continuação do desafio erosivo após os procedimentos restauradores permaneceu pouco observada. Além do mais, o artigo de número um trouxe também a investigação do desempenho do material frente a presença de outra substância, usada na tentativa de frear o avanço de tal desordem (HOOPER et al., 2014; SCHLUETER; KLIMEK; GANSS, 2013; ALGARNI, LIPPERT, HARA, 2015).

Os artigos acima fizeram uso de um protocolo que utilizou coca cola para simular as lesões erosivas, que se justifica por, além do fato dessa bebida ser consumida em grande escala, ter um alto potencial erosivo (RIOS et al., 2008) devido à um baixo pH e à baixa concentração de cálcio e flúor (FRANCISCONI et al., 2008). No que diz respeito ao uso de dentes bovinos, especialmente para avaliação de adesão aos tecidos dentais, essa prática está bem consolidada, visto que eles são capazes de produzir resultados semelhantes aos testes com dentes humanos (SOARES et al., 2016). Além do mais, o uso de incisivos bovinos permite uma

padronização da idade dos dentes utilizados e uma facilidade de obtenção em larga escala.

O primeiro estudo avaliou a influência do uso de um dentifrício anti-erosivo, contendo SnCl_2 , na resistência de união do adesivo Scotchbond Universal ao esmalte e à dentina submetidos a um desafio erosivo. O segundo artigo analisou a estabilidade de união do mesmo adesivo Universal frente ao desafio erosivo posterior à restauração, da mesma forma, em esmalte e dentina. Ambos estudos avaliaram o desempenho do material aplicado nas diferentes estratégias de condicionamento.

O primeiro artigo apresentado demonstrou que o uso do dentifrício anti-erosivo influenciou de formas diferentes os substratos esmalte e dentina, independentemente da estratégia utilizada. Houve uma redução da resistência de união do adesivo à dentina tratada, quando comparada à dentina hígida. Previamente, um estudo que avaliou a resistência de união à microtração à dentina tratada com um produto contendo estanho na composição, não encontrou diferença nos resultados entre dentina hígida e dentina tratada, nos espécimes onde a matriz orgânica desmineralizada não foi removida (FLURY, et al., 2013). É importante notar que, além de diferentes testes terem sido utilizados, o estudo em questão utilizou um adesivo autocondicionante de dois passos, sem aplicação ativa, que pode causar alteração na estrutura da matriz orgânica desmineralizada e isso pode ter trazido vantagens à adesão.

De acordo com estudos prévios, a adesão à dentina erodida é comprometida quando comparada à dentina hígida (CRUZ et al., 2015; FORGERINI et al., 2017; SIQUEIRA et al., 2018), o mesmo pôde ser observado na avaliação imediata desse estudo, quando a técnica de condicionamento ácido prévio foi utilizada. Diferentemente, quando utilizada a estratégia autocondicionante não foi possível observar diferenças significativas nos valores de resistência de união entre a dentina hígida e erodida, o que está de acordo com os resultados encontrados em uma pesquisa que utilizou o mesmo sistema adesivo (FRATTES et al., 2017).

Considerando o esmalte, os resultados encontrados em substrato hígido e erodido foram similares. O mesmo foi observado previamente, em um estudo realizado *in situ* que também utilizou coca cola como agente erosivo no protocolo (WANG et al., 2014). Por outro lado, o desempenho do material não foi prejudicado pelo tratamento nesse substrato, pelo contrário, foram obtidos maiores valores de resistência de união nesse substrato quando comparado ao esmalte hígido. Esse

resultado corrobora com o encontrado por Schlueter et al., 2013 que obteve maiores valores de resistência de união à microtração de um adesivo autocondicionante ao esmalte tratado com uma solução ácida de SnCl_2 (SCHLUETER et al., 2013a).

De acordo com a literatura existente, o uso da estratégia de condicionamento ácido prévio, em esmalte, é capaz de melhorar os resultados de resistência de união dos adesivos Universais (ANTONIAZI et al., 2016; DE GOES; SHINOHARA; FREITAS, 2014; HANABUSA et al., 2012; LOGUERCIO et al., 2015; MCLEAN et al., 2015; PERDIGÃO; LOGUERCIO, 2014; ROSA; PIVA; SILVA, 2015). O mesmo foi observado nesse estudo, independentemente da condição prévia ou do período de armazenamento avaliado.

No artigo de número dois, no qual foi avaliada a estabilidade união do adesivo Universal frente ao desafio erosivo posterior às restaurações, foram encontrados maiores valores de resistência de união à dentina quando o adesivo foi aplicado com condicionamento ácido prévio no grupo controle (armazenamento em saliva artificial – 7 dias), porém, não houve diferença entre as estratégias de aplicação quando os espécimes foram submetidos ao desafio erosivo. Dessa forma, é possível perceber que, considerando as estratégias de condicionamento, o modo de condicionamento ácido prévio sofreu maior influência, ou seja, foi mais sensível ao protocolo de erosão.

Considerando o fator armazenamento, o grupo de dentina que sofreu desafio erosivo foi negativamente afetado, tanto imediatamente após 24h quanto após seis meses de armazenamento. Essa redução dos valores de resistência de união está de acordo com um estudo prévio (TEDESCO et al., 2018), e podem ser atribuídos a uma desmineralização da dentina, nas regiões próximas à interface adesiva capazes de modificar, não somente o substrato (SALAS et al., 2011; WONGKHANTEE et al., 2006), mas também a interface adesiva em si.

Por outro lado, a resistência de união ao esmalte não foi comprometida pelo desafio erosivo, o que pode estar relacionado à estrutura desse substrato, o qual é mais mineralizado e tem menor conteúdo orgânico que a dentina. Além disso, a interface adesiva envolvendo o esmalte é mais estável (TEDESCO et al., 2018). Também foi possível observar valores mais elevados de resistência de união ao esmalte quando o adesivo universal foi utilizado com condicionamento ácido prévio, como mostrado em estudos anteriores (ANTONIAZI et al., 2016; DE GOES; SHINOHARA; FREITAS, 2014; HANABUSA et al., 2012; LOGUERCIO et al., 2015a;

MCLEAN et al., 2015; PERDIGÃO; LOGUERCIO, 2014; ROSA; PIVA; SILVA, 2015), reforçando o uso dessa técnica.

Ainda no que diz respeito ao substrato esmalte, o estudo apresentado não mostrou influência do armazenamento por seis meses na resistência de união do adesivo testado, independentemente da estratégia de aplicação utilizada. Essa maior estabilidade da adesão ao esmalte pode ser explicada pelo fato de que, em esmalte hígido, a degradação não é capaz de ocorrer tão rapidamente (VAN MEERBEEK et al., 2003). Da mesma forma, os desafios erosivos aos quais a interface adesiva em esmalte foi exposta também não influenciaram na resistência de união a esse substrato, o que pode estar relacionado à simplicidade e previsibilidade de adesão ao esmalte (PASHLEY et al., 2011; PERDIGÃO, 2010).

Assim, em ambos os artigos foi possível observar diferentes comportamentos relacionados ao esmalte e dentina, fato que era esperado tendo em vista as diferenças histológicas e morfológicas desses substratos. De qualquer forma, é possível notar a importância da avaliação do substrato no qual o sistema adesivo universal pretende ser aplicado, a fim de fazer a melhor escolha no que diz respeito à estratégia de aplicação a ser utilizada.

5 CONCLUSÃO

Com base nos artigos inclusos na presente tese, é possível concluir que existe diferença na performance e longevidade do sistema adesivo Scotchbond Universal aplicado em diferentes substratos (esmalte e dentina), e ainda, que tal desempenho é influenciado pelas modificações às quais esses substratos estão sujeitos no ambiente bucal. No que diz respeito ao efeito da modificação do substrato, prévio aos procedimentos restauradores, em dentina, o desafio erosivo reduz a resistência de união na estratégia de condicionamento ácido prévio, enquanto o tratamento com dentifrício anti-erosivo prejudica a adesão, independentemente da estratégia de condicionamento utilizada. No entanto, a resistência de união ao esmalte não é comprometida pela erosão ou pelo tratamento. Além disso, após a realização dos procedimentos restauradores, o desafio erosivo compromete a adesão do adesivo testado à dentina no modo de condicionamento ácido prévio, enquanto a resistência de união ao esmalte não é afetada.

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ANEXO A - NORMAS PARA PUBLICAÇÃO NO PERIÓDICO *THE 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
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- 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.

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2. Submission via e-mail as a PC-word document (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)

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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.
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REFERENCES

- **All references must be cited** in the text, according to the alphabetical and numerical reference list.
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Journal reference style:

1. Turp JC, Kowalski CJ, Stohler CS. Treatment- seeking patters 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:
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Legends—Figure legends should be grouped on a separate sheet and typed double-spaced.

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- Each table should be logically organized, on a separate sheet, and numbered consecutively.
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