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

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ESTUDO DO EFEITO DE TRÊS MÉTODOS DE ENVELHECIMENTO NA RESISTÊNCIA DE UNIÃO DO ESMALTE E DENTINA

Santa Maria, RS
2018

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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. Dr. Alexandre Henrique Susin

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2018

Teixeira, Gabriela
Estudo do efeito de três métodos de envelhecimento na resistência de união do esmalte e dentina / Gabriela Teixeira.- 2018.
47 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, 2018

1. Odontologia 2. Biomateriais 3. Dentística 4. Adesão 5. Envelhecimento artificial I. Susin, Alexandre Henrique II. Título.

Sistema de geração automática de ficha catalográfica da UFSM. Dados fornecidos pelo autor(a). Sob supervisão da Direção da Divisão de Processos Técnicos da Biblioteca Central. Bibliotecária responsável Paula Schoenfeldt Patta CRB 10/1728.

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Aprovado em 29 de maio de 2018:

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(Presidente da Banca/Orientador)

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Luiz Felipe Valandro Soares, Dr. (UFSM)

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Gabriel Kalil Rocha Pereira, Dr. (IMED)

Santa Maria, RS
2018

AGRADECIMENTOS

À minha família, por apoiarem todas as minhas escolhas e pelo suporte de sempre, graças a vocês estou concluindo mais uma etapa hoje. Obrigada por sonharem junto comigo!

À minha mãe, em especial, por ter sido minha maior incentivadora a vida inteira. Por ter confiado e enfrentado todas as minhas lutas ao meu lado. Por ser minha melhor amiga, cúmplice e a melhor mãe desse mundo. Tu és meu maior exemplo de professora, espero um dia ser metade do que te tornaste.

Ao meu namorado, Bruno, por todo apoio e compreensão. Obrigada por ser meu companheiro de vida, por sempre estar ao meu lado, me impulsionando nos momentos difíceis e comemorando minhas vitórias.

Às amigas Nicássia, Vitória, Patrícia e Samantha, meus presentes do mestrado. Obrigada pelo apoio, conselhos e companheirismo.

À minha amiga Laura, por todos esses anos de amizade. Obrigada por ter me ajudado tanto durante o mestrado, pelo incentivo, pelas risadas e comilanças.

À Danielle e a Bruna, por terem se tornado minhas amigas, além de colegas de pós-graduação. Obrigada por estarem sempre dispostas a ajudar, pelos conselhos e motivação.

Aos colegas de laboratório, pela convivência, pelos momentos de descontração e a troca de conhecimentos.

*Ao meu orientador, Prof. Alexandre Henrique Susin,*pelo convívio e por todas as oportunidades com que me presenteou. O mestrado superou todas as minhas expectativas e espero ter correspondido as suas também. Obrigada pela confiança, pelos ensinamentos e por ter se dedicado tanto ao meu trabalho.

À coordenação e professores do PPGCO, pela oportunidade, suporte e ensino que contribuíram para que eu alcançasse meu objetivo.

À Universidade Federal de Santa Maria, pela acolhida e por me proporcionar uma graduação e pós-graduação de alto nível.

À CAPES (Coordenação de Aperfeiçoamento de Pessoal de Nível Superior), pela concessão de bolsa de estudos.

RESUMO

ESTUDO DO EFEITO DE TRÊS MÉTODOS DE ENVELHECIMENTO NA RESISTÊNCIA DE UNIÃO DO ESMALTE E DENTINA

AUTORA: Gabriela Simões Teixeira
ORIENTADOR: Alexandre Henrique Susin

Avaliar os efeitos de três métodos de envelhecimento artificial (armazenamento em água, termociclagem e armazenamento em NaOCl) na degradação de um adesivo universal, no esmalte e na dentina. Noventa e seis terceiros molares humanos hígidos seccionados mesio-distalmente no terço médio, polidos com lixa 600 SiC e divididos de acordo com o método de envelhecimento: armazenamento em água, termociclagem e armazenamento em NaOCl. Um adesivo universal (Scotchbond Universal) foi aplicado em esmalte e dentina, no modo autocondicionante e condicionamento total. Matrizes de amido foram utilizados para realizar restaurações cilíndricas de resina composta com área de 1mm². Os espécimes foram submetidos ao teste μSBS. Os valores de μSBS em MPa foram submetidos a ANOVA de três fatores e teste de Tukey (p <0,05). Análise Weibull também foi realizada para estimar a probabilidade de falha. A análise de variância de três fatores revelou que o substrato (p= 0,00) e o protocolo de envelhecimento (p <0,00) tiveram efeito significativo sobre a resistência de união, mas o fator adesivo (protocolos) não foi significativamente diferente (p= 0,27). A distribuição de Weibull apresentou o maior módulo *m* no grupo de SH5h no condicionamento total da dentina, enquanto no esmalte, o maior *m* foi apresentado no grupo controle W24h, também no protocolo condicionamento total. Não foram observadas reduções significativas na resistência adesiva dos adesivos universais após os métodos de envelhecimento artificial. No entanto, a resistência de união do grupo esmalte, com protocolo de autocondicionamento, foi reduzida pela termociclagem. Os grupos foram insensíveis ao armazenamento de água a longo prazo e aumentaram os valores com imersão em solução de NaOCl.

Palavras-chave: Adesivo universal. Degradação. Envelhecimento artificial.

ABSTRACT

A STUDY OF THE EFFECT OF THREE AGING PROTOCOLS ON BOND STRENGTH ON ENAMEL AND DENTIN

AUTHOR: Gabriela Simões Teixeira
ADVISOR: Alexandre Henrique Susin

To evaluate the effects of three artificial aging methods (water storage, thermocycling and NaOCl storage) on the degradation of a universal adhesive, in enamel and dentin. Ninety–six non-carious human third molars were sectioned mesio-distally at the middle third, polished with 600-grit SiC and divided according to the aging method: water storage, thermocycling and NaOCl storage. A universal adhesive (Scotchbond Universal) was applied in enamel and dentin, on self-etch and etch-and-rinse mode. Starch tubing was used as matrix to perform 1mm² of area of cylindrical composite resin restorations. The specimens were submitted to μSBS test. The μSBS values in MPa were subject to three-way ANOVA and post hot Tukey test ($p<0.05$). Weibull Analysis was also performed to estimate the probability of failure. Three-way ANOVA revealed that substrate ($p=0.00$) and aging protocol ($p<0.00$) had significant effect on bond strength, but the factor adhesive was not significantly different ($p=0.27$). Weibull distribution presented the highest modulus *m* in the group of SH5h on etch-and-rinse on dentin while on enamel, the highest *m* was presented in the control group W24h, also on etch-and-rinse mode. No significant reductions in SBSs of universal adhesives were observed after methods of artificial aging. The bond strength of enamel group, with self-etch protocol, was reduced by thermocycling. In contrast, the groups were insensitive to long-term water storage and increased the values with storage in NaOCl solution.

Keywords: Artificial aging. Degradation. Universal adhesive.

SUMÁRIO

1 INTRODUÇÃO

O surgimento e desenvolvimento dos sistemas adesivos tem permitido uma adesão direta dos materiais restauradores com o substrato dentário (DENG et al., 2014), sendo a longevidade fator essencial para os quesitos biológicos e estéticos. Valores elevados de resistência de união são desejáveis, já que a interface de união deve suportar as tensões causadas pela contração de polimerização da resina composta e evitar falhas adesivas causadas pelas demandas térmicas, mecânicas e químicas do meio bucal (ROCHA et al., 2007).

No entanto, apesar das significativas melhoras dos sistemas adesivos, a interface permanece a área mais suscetível às falhas das restaurações de resina composta. Isso acontece porque sua durabilidade e estabilidade são afetadas pelas forças oclusais, hábitos alimentares, temperatura intraoral e ambiente úmido (DE MUNCK et al., 2003). Como consequência, tal degradação enfraquece a adesão e produz deteriorações marginais, as quais reduzem a longevidade da restauração. (AMARAL et al., 2007).

Por esses motivos, simulações *in vitro* podem contribuir como preditores da longevidade de materiais odontológicos, a partir da avaliação de suas características de deterioração mecânica e estrutural com o decorrer do envelhecimento clínico (MORRESI et al., 2014). O armazenamento em água destilada é o método mais recorrente em pesquisas, visto que a presença da água é determinante na deterioração da interface adesiva (AMARAL et al., 2007). Durante o armazenamento em água, a hidrólise promove a desintegração das fibras colágenas da camada híbrida e a resina associada na área de dentina desmineralizada (fibras colágenas desprotegidas) se reduz gradativamente ao longo do tempo (HASHIMOTO et al., 2003).

A temperatura intraoral varia de acordo com a alimentação, bebidas e os hábitos respiratórios, e tais mudanças rápidas de temperatura inevitavelmente afetam a estabilidade da restauração adesiva (MORRESI et al., 2014). Assim, a termociclagem é outro método de envelhecimento artificial comumente utilizado para avaliar a durabilidade da adesão. A ação da água quente pode acelerar a hidrólise de colágeno desprotegido e extrair oligômeros pouco polimerizados, e após, devido ao maior coeficiente de contração/expansão térmica do material restaurador (quando comparado com ao do tecido dentário), é gerada uma tensão de contração/expansão repetitiva na interface dente-biomaterial (DE MUNCK et al., 2005). Dessa forma, a alternância da imersão em água aquecida e resfriada gera tensões na interface adesiva semelhantes às produzidas no ambiente oral.

Nos últimos anos foi introduzida, como metodologia de envelhecimento, a imersão em solução de hipoclorito de sódio a 10% (NaOCl) com a finalidade de avaliar através de um método prático a durabilidade da adesão e sem demanda de maiores períodos de tempo (DENG et al., 2014), através da exposição e dissolução de fibras colágenas (DE MUNCK et al., 2007; YAMAUTI et al., 2003). O NaOCl a 10% tem um efeito proteolítico não específico, portanto degrada componentes orgânicos que não estão completamente protegidos na interface adesiva, testando assim indiretamente a capacidade dos monômeros resinosos de proteger a matriz colágena da dentina (YAMAUTI et al., 2003).

O presente estudo tem como objetivo comparar os efeitos dos três métodos citados no envelhecimento artificial da interface adesiva, em esmalte e dentina, utilizando um adesivo univesal, com teste de microcisalhamento. A hipótese nula é que a resistência de união não será significativamente afetada pelos métodos de envelhecimento em teste, independentemente do substrato dental e do modo de aplicação do adesivo.

2 ARTIGO - A STUDY OF THE EFFECT OF THREE AGING PROTOCOLS ON BOND STRENGTH ON ENAMEL AND DENTIN

Este artigo será submetido ao periódico *Dental Materials*, *Elsevier*, ISSN: 0109-5641, Fator de impacto = 4.070; Qualis A1. As normas para publicação estão descritas no Anexo A.

A study of the effect of three aging protocols on bond strength on enamel and dentin

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Declarations of interest: none.

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Abstract

Objectives: to evaluate the effect of three artificial aging methods (water storage, thermocycling and NaOCl storage) on the bond strength of a universal adhesive, in enamel and dentin. Methods: Ninety–six non-carious human third molars were sectioned mesio-distally at the middle third, polished with 600-grit SiC paper and divided according to the aging method: water storage, thermocycling and NaOCl storage. A universal adhesive (Scotchbond Universal) was applied in enamel and dentin, on self-etch and etch-and-rinse mode. Starch tubing was used to perform cylindrical composite resin restorations that were tested under μSBS test. Results: the μSBS values in MPa were subject to three-way ANOVA and post hot Tukey test ($p<0.05$). Weibull Analysis was also performed to estimate the probability of failure. Three-way ANOVA revealed that substrate ($p=0.00$) and aging protocol ($p<0.00$) had significant effect on bond strength, but the factor adhesive mode was not significantly different ($p=0.27$). Weibull distribution presented the highest *m* in the group of SH5h on etch-and-rinse on dentin while on enamel, the highest *m* was presented in the control group W24h, also on etch-and-rinse mode. Significance: no significant reductions in SBSs of universal adhesives were observed after methods of artificial aging. Nevertheless, the bond strength of enamel group, with self-etch protocol, was reduced by thermocycling. In contrast, the groups were insensitive to long-term water storage and increased the values with storage in NaOCl solution.

Keywords: Artificial aging. Degradation. Universal adhesive.

1. Introduction

The interface between composite resin and the cavity walls are the most critical region in bonded restorations. Unprotected collagen fibrils depleted [1] and chemical hydrolysis of the ester bonds are considered the main reasons for hybrid layer degradation. These processes occur concomitantly and definitively contribute to the reduction of bond strength, over time [2].

Composite resin restorations are highly dependent of bonding to enamel and dentin to adequately to seal the margins of the cavities and to retain the restorative material, preserving form and function as well as the longevity of the procedure.[3][4] Despite the advances in dental bonding over the past years the oral environment and dental tissues remain unfavorable to preservation of the effectiveness of the bonded interfaces on long term.[5][6] Adhesive systems act on different ways over enamel and dentin, since the tissues have different micromorphology and balance of organic and inorganic contents.[4] In the enamel the hybridization process occur easier and simpler than in dentin. The enamel's contents are highly homogeneous, stable and the micromorphology is proper to monomer impregnation. The dentin has a inhomogeneous nature and high organic content, and the effects of the degradation occur significantly faster.[7][8]

Studies in vitro concerning bonding degradation are widely used as a way of to predict clinical performances of bonded restorations[9]. Mechanical tests as microshear bond strength may be performed to test the influence of different aging methods on bond strength. The aging methods such as storage in water [10][11][12] thermocycling [13][14][15], storage in NaOCl solution [16][17][18] and two or more simultaneous methods, are used to simulating intraoral conditions and its repercussions in the bonding efficacy on long term of the adhesive systems.[17][19][20][21]

The aging by storage in water commonly use the immersion of the samples in distilled water at 37°C from 3[22] to 12 months [11] to partially simulate the oral environment conditions. In the thermocycling method from 500[23] to twenty thousand cycles [24], hot and cool water can simulate the hydrolysis of unprotected collagen fibrils by repetitive stress of contraction / expansion resulting in gaps propagation along the adhesive interface allowing water and pathogenic oral fluid penetration.[25][26][27] The method of storage in 10% of NaOCl solution from 1 or 5h[17][27]uses the properties of non-specific proteolytic effect of the hypochlorite to degrade the organic resin and tooth interface components - as unprotected collagen fibrils -to simulate the aging effect [16][17][18][27].

To the best of authors' knowlegde, there is limited information about aging methods and its effect on enamel and dentin. Also, taking into acount the importance to predict the degradation of bonded interfaces and its importance for clinical performance the resin composite restorations, it is relevance to assess the mechanical tests performed in groups of samples which were submitted to different aging methods in the same study, since can to help researchers elucidate how each one of the methods can influence in the choice of aging protocols for subsequent studies.

Therefore, the present study aims to compare the effects of three artificial methods of aging of bonded interface in enamel and dentin of a universal adhesive, under microshear bond strength test. The null hypothesis is that the bond strength is not significantly affected by any aging methods independently of the substrate and adhesive approaching.

2. Materials and methods

2.1. Study design

This in vitro study was approved by the Local Ethics Committee in Research (protocol 2.054.447). Ninety–six non-carious human third molars were collected from an institucional bank of the teeth, up to 3 months after extraction. The teeth were stored in 0.5% chloramine solution at 37ºC during 7 days for disinfection. All tests were performed by a single blinded operator.

2.2. Specimens preparation

The roots were removed, and the crowns were sectioned mesio-distally at the middle third using a low-speed water cooled diamond saw in a cutting machine (Labcut 1010, ExtecInc, Enfield, CT, USA). The sections were embedded in pvc rings with self-curing acrylic resin (JET, Classico Art Od, São Paulo, Brazil). The slices with exposed enamel and dentin had standardized the smear layer using 600-grit silicon carbide paper for 1 minute in a circular mechanical polishing machine (EcoMet 250, Buehler, Lake Bluff, IL, USA) under water irrigation.[28]

2.3. Bonding and restorative procedures

A universal adhesive (Scotchbond Universal – 3M ESPE Dental Products, St Paul, MN, USA) on self-etch and on etch-and-rinse mode, was applied according manufacturer instructions in enamel and dentin. Before adhesive photo-curing, three (3) starch tubes (Renata, PastificioSelmi, Londrina, PR, Brazil) of 1 mm of high and 0.96 mm of internal diameter were positioning over each substrate [29]. A light-emitting diode (Emitter D – Shuster Eq. Od. Ltd, Santa Maria, Brazil) with 900 mW/cm^2 light output was used to photo-cure adhesive for 10s. Resin composite (Filtek Z350 XT shade A2, 3M ESPE Dental Products, St. Paul, MN, USA) was used to fill in into the starch tubes. All adhesive procedures and restorations were performed by a trained operator in a room with controlled temperature and humidity. The Table 1 shows the techniques used on the bonding and restorative procedure.

After 24 h of water storage the starch matrix were removed with air-water spray and the interfaces enamel/resin and dentin/resin were examined using a stereomicroscope at 10X of magnification (Discovery V20, Zeiss; Oberkochen, Germany). Specimens that presented bonding defects were discarded and replaced.

The samples were randomized divided into thirty-two (n=6) according the mode of the adhesive was used (2– etch-and-rinse or self-etch mode), substrate (enamel and dentin) and aging methods (8): control, (no aging – the samples were tested after 24 h); 37ºC water storage (6 m and 12m); themocycling (10.000x, 20.000x, 30.000x) and 10% sodium hypochlorite storage (1 h and 5 h) - Table 2.

2.4. Microshear Bond Strength Test

After the samples had been submitted to the aging process, a blinded operator performed the tests of microshear bond strength in enamel and dentin by wire loop technique. [30] A 0.2 mm stainless steel wire was attached to a specific device in a universal testing machine (EMIC DL 1000, Instron Brazil, S.J.Pinhais, Brazil) and shear load was applied at crosshead speed of 1.0 mm/min, with cell of 1kN, until the failure of the bonded restoration. Care was taken to keep the wire loop adjusted to the adhesive interface of the pin restoration before starting the test.

2.5. Failure Mode

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

2.6. Statistical analysis

The experimental unit in this study was the tooth. The average of the three measures, in MPa, were assumed as the measured value for each tooth and used for purposes of Kolmogorov-Smirnov test, three-way ANOVA and Tukey's test. The Kolmogorov-Smirnov was used for to check the normality of the data followed by a three-way ANOVA with the factors: adhesive approach (2), substrate (2) and aging method (8), and the post hoc Tukey's test, for multiple comparisons.

A Weibull Analysis was also performed to estimate the probability of failure (Pf). Since that to perform a robust Weibull analysis the number of specimens need to be at least 10 (ten) [31] the three measures of bond strength performed on each substrate was considered as individual values. Thus, for Weibull analysis purposes, each group had eighteen measures. The Weibull statistical analysis measures the parameters scale (σ) and the shape (Weibull modulus - *m*) for each group of study separately. In this analysis the left and right-censored values was not established as it was done by El-Damanhoury&Gaintanaopoulou, 2015, since all specimens which presented pre-testing failures were substituted and there is no a pre-defined criterion for determining which values are the lowest or the highest ones (left and right-censored, respectively)[32]. In this case to conduct the Weibull analysis, the software suggest for researchers to consider the censoring value as 0 (zero), in accordance with previous studies using this value as normalizing a parameter [33][34].

3. Results

The μSBS, expressed in MPa, and standard deviations are summarized in Table 3. Three-way ANOVA revealed that substrate (p=0.00) and aging protocol (p<0.00) had significant effect on bond strength. Considering the factor adhesive mode (as self-etch or etch-and-rinse) was not significantly different, p=0.27.

The triple interaction (adhesive x substrate x aging protocol) had not statistical significant effect, p=0.47. The general mean of bond strength on enamel was 13.18 MPa while on dentin, the mean was 18.14 MPa. The interactions between factors in pairs revealed significant effect. The interaction between substrate and protocol had p=0.01.

The higher value of bond strength considering the protocols of aging, independent of adhesive approaching and substrate was 19.82 MPa presented by the group of aging by hypochlorite for 5 h (SH5h) while the lower value was 13.84 from 30 thousand times of thermocycling (T3) group. None group presented significantly lower result than the baseline (W24h), independently of substrate or adhesive approaching.

Considering the self-etch mode on dentin substrate it was observed that the storage in water for 6 months (W6m) presented higher results in bond strength than the baseline. On the other hand, W12m group presented result not significantly different of the baseline and the W6m group.

The thermocycling negatively affected the microshear bond strength to adhesive on self-etch mode on dentin substrate, presenting lower results than the others. On the hypochlorite groups, SH1h and SH5h, the lower results were observed on the self-etch mode on enamel, 12,56MPa and 13,16MPa, respectively.

For all groups, regardless of the substrate, adhesive approaching and aging, adhesive/mixed failures were predominant (Table 4). Specimens that had premature failures were replaced.

Table 5 summarizes Weibull distribution presented the highest *m* in the group of SH5h on etch-and-rinse (5.42) on dentin while on enamel, the highest *m* was presented in group W24h (5.48), also on etch-and-rinse mode. It was observed that groups of enamel and dentin as well as the groups of self-etch and etch-and-rinse did not present a defined pattern of scale and shape, coherently with ANOVA that presented very distributed significance between the groups (Table 3).

In the graphs of the figure 1(A, B and C) are presented the probability of failure over time (probability plot) results of enamel and dentin separately, for each aging method (presenting in the same graph all time of ageing – for water aging and all number of cycles – for thermocycling and sodium hypochlorite aging).

Under this view it was noticed that the aging with sodium hypochlorite presented more predictable results in both, enamel and dentin, since it`s values of shape and confidence interval (also called as confidence band) were higher than the other aging methods.

4. Discussion

Despite of the adhesive systems have been *in vitro* widely studied in their bonding properties on both, enamel and dentin substrates, the use of the results as predictive applying still remains as a doubt to the researchers [35][36][37]. It is suggested that the mechanism of bond degradation observed in laboratory techniques may not be applied at the same rate clinically or the bonds have a secondary role in the clinical success of bonded restorations [36]. There are still disagreements over *in vitro* aging methods that can predict the possible behavior of the adhesive-substrate interface under similar clinical conditions [17][38].

In this present study, the adhesive approaching had no affect significantly the bond strength, conversely the substrate and aging protocol. Thus the null hypothesis was partially rejected. None group reduced μSBS compared to baseline. Consistently with our findings, it was reported that adhesive remained stable on aging since the methods used did not change significantly the bond strength [26][38][39].

4.1. Thermocycling

Thermocycling is widely used in dental research, to evaluating the performance of adhesive restorations [17]. During temperature changes, the difference in thermal expansion between the resin and the tooth substrate promote volumetric changes that result in fatigue and gaps along of the adhesive-dentin interfaces [26]. According the International Organization for Standardization (ISO TR 11450) to simulate the long term challenging of bond durability of dental biomaterials the samples shall be submitted a 500 cycles through water baths at 5ºC and 55ºC [40]. Usually, each study arbitrarily determines the number of thermocycles, since ISO-based research protocols concluded that the number of 500 cycles are very limited to the thermal changes promote effect on bond strength or microleakage of adhesive interface. [25][27][41][42].

The efficacy of the thermocycling test in evaluating bond strength already been questioned [25][43], and the low C-factor of the specimens could have diminished the expansion/contraction stress effect [44]. Actually it is accept that approximately 10,000 thermocycles correspond to 1 year in vivo degradation [25]. Therefore, in this study, the bond strength retained by Scotchbond Universal, on both strategies that can be comparable at least with 3 years of clinical use, which we may be considered as a reasonable aging time.

In general, the values on dentin after thermocycling to µSBS were higher than enamel, especially for self-etch protocol. Nevertheless, corroborating study of Hariri et al [42], the aging with thermocycling presented more predictable enamel results, since that values of shape, *m* and confidence interval, to etch-and-rinse protocol, showed more adequate.

Although 20.000 and 30.000 cycles of thermocycling resulted in the worst results of µSBS, these values were not significantly lower than the baseline, supporting other studies with 5.000 [43], 20.000 [26] and 30.000 [38] thermocycles that did not significantly affected the bond strength. Saboia et al.[41] showed that thermocycling reduced the bond strength when 60.000 cycles was used, suggesting that the number of cycles employed in this research was not sufficient to affect the bonding stability. Another issue to be considered is that the values obtained in these studies were based on microtensile test. Hence, it is hypothesized that the baseline of the microshear test usually presents lower values because, supposedly, it needs a time greater than 24h to become stable, thus justifying that none number of thermocycles promoted worse than the control group.[45]

4.2. Water Storage

The most frequent aging method related on the literature is water storage under at room temperature or 37ºC.[11][22][46][47] It has generally been accepted that the water aging method for dental materials should be a way of safely predict the time in which an adhesive interface would remain stable.[4]

Studies carried out with water storage indicate that degradation was accelerated by hydrolysis of hydrophilic resin components and by host-derived proteases with collagenolytic activity [2]. Conversely, this method did not affect the adhesive strength, independently of the time of aging, substrate or protocol. The storage for 1 year presented result not significantly different of the baseline and the W6m group. Other researches corroborates these results, suggesting that bond strength with universal adhesives remains stable even after 1 year of water storage, especially in self-etch mode [38][48].

Based on that, it is evident that the effects are also dependent on the particularly the adhesive tested [49] or used protocol. For self-etch approaching, the adhesive infiltrates around collagen network and dentin inorganic components, developing to form a regular hybrid layer [50], which may explain the result of dentin after 6 months. With the incorporation of hydrophilic and ionic resinous monomers in the adhesion, as in simplified adhesives, the formation of hybrid layers behaves like semi permeable membranes that allow the movement of water throughout the adhesive interface even after the polymerization [51].

According Scherrer et al.[21], the behavior of these adhesives bonded to dentin are better evaluated using the Weibull distribution function, predicting the likelihood of failure at specific stress levels, in which Weibull *m* report the variability of the results. The lowest *m* value was reached in dentin group on the etch-and-rinse protocol, after 6 months. In addition, the *m* values after 1 year of aging in enamel group were also low. This demonstrated a low reliability, despite being inside of the confidence interval.

Several inconclusive studies are related by Hentzie et al. concerning to the difficult to reproduce oral conditions and design a fatigue method that corresponds to the situation *in vivo* in addition to the differences between enamel and dentin tissues.[52] Even so this study found that the bond strength was more influenced by the approaching of the adhesive and by the region tested (enamel or dentin) independently of the time of storage. It is in accordance with studies of Tsuchiva et al. and Takahashi et al.[53][54].

Regional variability of dentin tissue and, more recently, chemical bonding component of dental adhesives were added to the model of study.[55][56][57] It was observed that contemporary adhesives follow the tendency of having in their formulation the MDP (10-methacryloyldecyl dihydrogen phosphate) as functional monomer.[53] In this study it was chosen to use only Scotchbond Universal as adhesive system, despite the two approaching (etch-and-rinse and self-etch modes), once the study was focused in the effect the aging methods. The obtained results in the groups of W24, W6m and W12m generally did not present statistically differences, except to dentin substrate on the self-etch mode to W24 and W6m. At Weibull analysis it was not relevant since these groups reached similar values of *m*, 4.51 and 4.45, respectively. The Weibull *m* is a value that show the distribution of defects based on the confidence intervals from scale. It is correct to state that high values of *m* indicate narrow distribution of defects while lower values of *m*, indicate that the defects occurred irregularly. This can be assumed that the cited groups presented showed a regular distribution of defects.

4.3. Storage in 10% NaOCl

Unlike others studies [17][41], storage in 10% NaOCl solution did not reduce the bond strength. These studies used the same concentration of the NaOCl solution, but different adhesives and bond strength test. In the other hand, the Weibull *m* showed higher values in dentin with etch-and-rinse protocol, in the SH5h, indicating lower variability (that is, lower spread) in the bond strength to dentin, which means high reliability of the characteristic bond strength and probable absence of critical flaws [21].

This may have been a result of the incorporation of the 10-MDP monomer in the adhesive, that provide acidity and self-etching capability. Even though at differing levels, MDP-containing adhesives form nano-layers at the adhesive interface, depending on the formulation of the adhesive. Stable MDP-Ca salt deposition together with these nano-layers may explain the high stability of MDP-based bonding [58]. Other variables in the composition can be explained by the stability of the Scotchbond Universal, for instance, the presence of the polyalkenoic acid copolymer (PAC), as it is used in resin-modified glass ionomer Vitrebond (3M ESPE), that also form bonds chemically and spontaneously to hydroxyapatite.[59]. Yoshida et al.[58] suggested that PAC could compete with the MDP, however studies have shown that the presence of PAC promote higher bond strength than a PAC-free adhesive with the similar composition [39][60].

This findings support that Scotchbond Universal remains stable after artificial aging methods. However, this is an *in vitro* study, so further clinical trials are needed to better understanding the behavior of bonded restorations using universal adhesives.

5. Conclusion

No significant reductions in μSBSs of universal adhesive were observed when submitted to the artificial methods of aging, i.e., the bond strength was not affected in the majority of the groups.

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

6. References

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Table 1 - Materials used in this study – application procedure

Adhesive system – Manufacturer and batch number	**Type**	**Composition**	**Self-etch mode**	**Etch-and-rinse mode**
ScotchbondUniversal – 3MESPE Dental Products, St Paul, MN, USA, ________	Adhesive System	2-HEMA, 10-MDP, dymethacrylate resins, Vitrebond™ copolymer, silane, filler, ethanol, water, initiatiors	1. Apply the adhesive to the prepared tooth agitating for 20s 2. Gently air-dry for 5s 3. Light cure for 10s	1. Apply phosphoric acid gel for 20s, 2. Rinse with water, 3. Dry with cotton pellets, 4. Apply adhesives for the self-etch mode
Filtek Z350 – 3M ESPE Dental Products, St Paul, MN, USA	Resin Composite	Silica 20nm, zirconia 4-11nm, Bis-GMA, Bis-EMA, UDMA,TEGDMA	1.Apply two increments of resin composite 2. Light cure for 20s	

Abbreviations: 2-HEMA, 2-hydroxyethyl methacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogenphosphate.

Table 2. Adhesive Approaching / Aging methods - (grouping n=6)

Adhesive approcaching	**control - 24 hs in water**	**water storage - 6m**	**water storage - 12m**	**thermocycling 10000 x**	**thermocycling 20000 x**	**Thermocycling 30000 x**	**NaOCl 10% - 1h**	**NaOCl 10% - 5 h**
Scotchbond Universal etch-and-rinse mode	The samples were prepared and immediately stored in distiled water for 24 h before testing	The samples were kept immersed in water at 37°C for 6 months before testing. Water was changed weekely	The samples were kept immersed in water at 37°C for 12 months before testing. Water was changed weekely	The samples were thermocycled for 10000 times at 5°C to 55°C for 30 s each batch - 5 s of transfer time	The samples were thermocycled for 20000 times at 5°C to 55°C for 30 s each batch - 5 s of transfer time	The samples were thermocycled for 30000 times at 5°C to 55°C for 30 s each batch - 5 s of transfer time	The samples were kept immersed in 10% NaOCL solution at room temperature for 1 h	The samples were kept immersed in 10% NaOCL solution at room temperature for 5 h

Adhesive approcaching	**control - 24 hs in water**	**Water storage - 6m**	**Water storage - 12m**	**thermocycling 10000 x**	**thermocycling 20000 x**	**Thermocycling 30000 x**	**NaOCl 10% - 1h**	**NaOCl 10% - 5 h**
Scotchbond Universal Self-etch mode	The samples were prepared and immediately stored in distiled water for 24 h before testing	The samples were kept immersed in water at 37°C for 6 months before testing. Water was changed weekely	The samples were kept immersed in water at 37°C for 12 months before testing. Water was changed weekely	The samples were thermocycled for 10000 times at 5°C to 55°C for 30 s each batch - 5 s of transfer time	The samples were thermocycled for 20000 times at 5°C to 55°C for 30 s each batch - 5 s of transfer time	The samples were thermocycled for 30000 times at 5°C to 55°C for 30 s each batch - 5 s of transfer time	The samples were kept immersed in 10% NaOCL solution at room temperature for 1 h	The samples were kept immersed in 10% NaOCL solution at room temperature for 5 h

Table 3 – Microshear bond strength, standard deviation and statistical significances

	Scotchbond Universal Self-etch mode		**Scotchbond Universal Etch-and-rinse mode**	
Aging method/ Substrate	**E**	**D**	**E**	**D**
W24h	12.03(2.16) ^{Aa}	16.13 (2.05) ^{Ab}	15.53 (1.58) ^{Aa}	13.43 (3.40) ^{Ac}
W6m	12.07(1.04) ^{Ba}	23.66 (4.17) ^{Aa}	11.49 (3.39) ^{Ba}	11.22 (3.88) ^{Bc}
W12m	11.91(3.02) ^{Aa}	18.52 (3.65) ^{Aab}	13.81 (3.30) ^{Aa}	14.26 (4.61) ^{Ac}
T10000	10.21(1.31) ^{Ca}	20.26(3.45) ^{ABab}	15.63 (3.27) ^{BCa}	16.34 (5.39) ^{BCbc}
T20000	7.00 (1.79) ^{Ba}	16.38 (4.87) ^{Ab}	17.73 (2.43) ^{Aa}	16.17 (3.75) ^{Abc}
T30000	8.26 (2.12) ^{Ba}	17.41 (2.38) ^{Ab}	14.66 (3.86) ^{ABa}	15.06(3.80) ^{ABbc}
SH1h	12.56(1.91) ^{Ba}	20.98 (5.94) ^{Aab}	17.13 (3.55) ^{ABa}	21.90 (4.48) ^{Aab}
SH5h	13.16(1.40) ^{Ca}	25.67 (3.24) ^{Aa}	17.67 (4.41) ^{BCa}	22.79 (3.35) ^{ABa}

The same small letter in columns indicate no statistical differences. Different capital letter in lines indicate statistic significant difference (α0.05)

Table 4 – Failure Mode

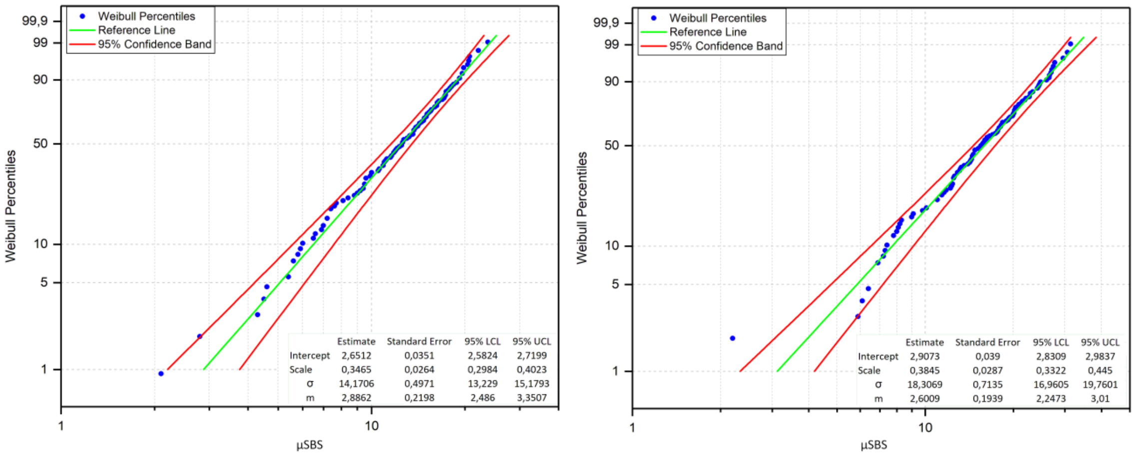
Groups	**Failure mode (%)**			
	Adhesive/ Mixed	Cohesive in enamel	Cohesive in dentin	Cohesive in resin
Control	100%	-	-	-
Storage in water	80%	5%	15%	-
Thermocycling	86%	4%	10%	-
Storage in NaOCl	100%	-	-	-

Table 5 - Estimate Weibull value - scale= “σ” and shape= *m* - [Standard Error], lower “LCL” and upper “UCL” confidence level at α=0.05 and results of microshear bond strength “μSBS” in MPa (Standard Deviation) of experimental groups.

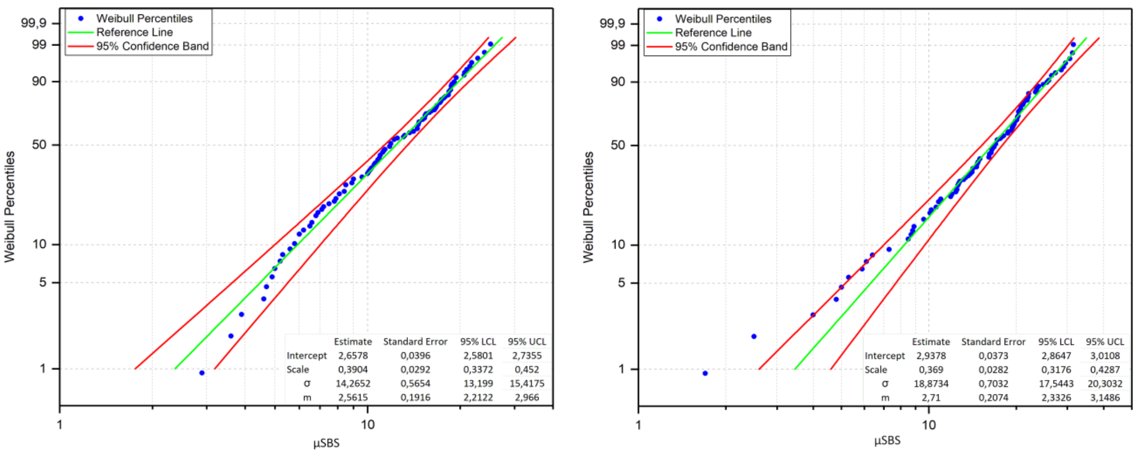
Aging Method	**SBU - Adhesive Approaching**	**enamel**							**dentin**						
		σ			***m***				**σ**			***m***			
		Estimate Weibull Characteristic strength	**LCL**	**UCL**	**Estimate**	**LCL**	**UCL**	**μSBS (SD)**	**Estimate Weibull Characteristic strength**	**LCL**	**UCL**	**Estimate**	**LCL**	**UCL**	**μSBS (SD)**
W24h	Self-etching	13.37 [0.75]	11.77	15.18	3.80 [0.75]	2.58	5.61	12.03 (2.16)	17.00 [3.94]	15.25	18.96	4.51 [0.76]	3.24	6.28	16.13 (2.05)
	Etch-and-Rinse	16.85 [0.76]	15.42	18.42	5.48 [1.03]	3.79	7.93	15.53 (1,58)	14.90 [0.99]	13.06	16.99	3.70 [0.69]	2.56	5.33	13.43 (3.40)
W6m	Self-etching	13.32 [0.83]	11.78	15.05	3.96 [0.72]	2.77	5.68	12.07 (1.04)	25.88 [1.44]	23.19	28.88	4.45 [0.78]	3.15	6.28	23.66 (4.17)
	Etch-and-Rinse	13.02 [1.41]	10.52	16.12	2.29 [0.40]	1.62	3.25	11.49 (3.39)	11.66 [1.49]	9.07	14.99	1.94 [0.34]	1.37	2.74	11.22 (3.88)
W12m	Self-etching	12.21 [1.34]	9.85	15.14	2.25 [0.42]	1.55	3.26	11.91 (3.02)	21.42 [1.47]	18.71	24.52	3.58 [0.67]	2.47	5.17	18.52 (3.65)
	Etch-and-Rinse	15.56 [1.42]	13.00	18.62	2.70 [0.52]	1.85	3.94	13.81 (3.30)	17.11 [1.38]	14.61	20.05	3.08 [0.58]	2.12	4.46	14.26 (4.61)
T10000	Self-etching	11.60 [0.61]	10.45	11.86	4.63 [0.92]	3.13	6.86	10.21 (1.31)	22.30 [1.37]	19.77	25.15	4.05 [0.71]	2.86	5.73	20.26 (3.45)
	Etch-and-Rinse	17.10 [0.88]	15.45	18.93	4.77 [0.90]	3.28	6.92	15.63 (3.27)	18.26 [1.47]	15.58	21.40	3.06 [0.56]	2.13	4.39	16.34 (5.39)
T20000	Self-etching	8.71 [0.69]	7.44	10.18	3.11 [0.59]	2.14	4.52	7.00 (1.79)	17.43 [1.37]	14.93	20.34	3.14 [0.60]	2.15	4.59	16.38 (4.87)
	Etch-and-Rinse	19.43 [1.16]	17.28	21.85	4.17 [0.71]	2.98	5.82	17.73 (2.43)	18.32 [2.45]	14.09	23.81	1.84 [0.36]	1.25	2.71	16.17 (3.75)
T30000	Self-etching	9.52 [0.68]	8.28	10.96	3.47 [0.63]	2.42	4.91	8.26 (2.12)	19.44 [1.49]	16.72	22.61	3.21 [0.59]	2.23	4.63	17.41 (2.38)
	Etch-and-Rinse	17.30 [1.17]	15.14	19.76	3.64 [0.69]	2.51	5.28	14.66 (3.86)	16.86 [1.71]	13.82	20.57	2.44 [0.43]	1.71	3.47	15.06 (3.80)
SH1h	Self-etching	13.94 [1.00]	12.10	16.06	3.45 [0.59]	2.46	4.85	12.56 (1.91)	23.00 [1.67]	19.93	26.54	3.33 [0.66]	2.25	4,91	20,98 (5.94)
	Etch-and-Rinse	18.97 [1.23]	16.70	21.54	3.82 [0.71]	2.65	5.51	17.13 (3.55)	23.49 [1.54]	20.65	26.72	3.76 [0.73]	2.57	5.51	21.9 (4.48)
SH5h	Self-etching	14.25 [0.67]	13.00	15.61	5.32 [0.93]	3.76	7.51	13.16 (1.40)	28.04 [1.49]	25.26	31.12	4.68 [0.84]	3.29	6.64	25.67 (3.24)
	Etch-and-Rinse	19.52 [1.15]	17.39	21.91	4.20 [0.82]	2.85	6.17	17.67 (4.41)	24.67 [1.13]	22.54	27.00	5.42 [0.96]	3.83	7.68	22.79 (3.35)

Figure 1 – Probability plot with Weibull curves at maximum likelihood estimation. Estimate Scale (σ) and Shape (m) values with Standard Error and Lower Confidence Level (LCL) and Upper Confidence Level (UCL). The Confidence Interval (95% CI) is the band between LCL and UCL.

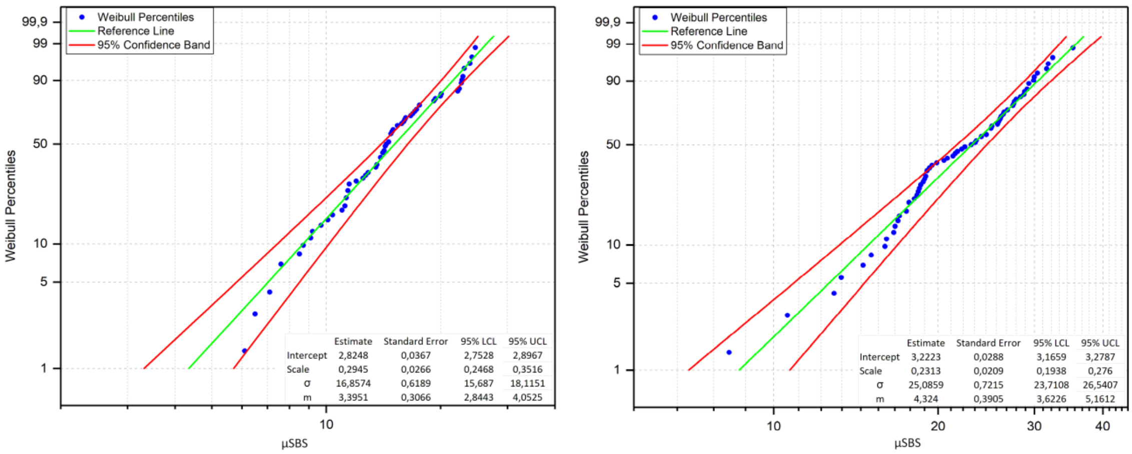
A)



B)



C)



A) Groups aged in water 24h, 6m and 12m, (SBU SE and ER), enamel and dentin, respectively. B) Groups thermocycled, 10000, 20000 and 30000 times, (SBU SE and ER), enamel and dentin, respectively. C) Groups aged in sodium hypochlorite, 1h and 5h, (SBU SE and ER), enamel and dentin, respectively.

3 CONCLUSÃO

Não foram observadas reduções significativas na resistência de união do sistema adesivo universal submetido a métodos de envelhecimento artificiais. Com isso, a hipótese nula foi parcialmente rejeitada.

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[1] Van der Geer J, Hanraads JAJ, Lupton RA. The art of writing a scientific article. J Sci Commun2010;163:51–9.

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[2] Strunk Jr W, White EB. The elements of style. 4th ed. New York: Longman; 2000.

Reference to a chapter in an edited book:

[3] Mettam GR, Adams LB. How to prepare an electronic version of your article. In: Jones BS, Smith RZ, editors. Introduction to the electronic age, New York: E-Publishing Inc; 2009, p. 281–304.

Reference to a website:

[4] Cancer Research UK. Cancer statistics reports for the UK, ttp://www.cancerresearchuk.org/aboutcancer/statistics/cancerstatsreport/; 2003 [accessed 13 March 2003].

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[dataset] [5] Oguro M, Imahiro S, Saito S, Nakashizuka T. Mortality data for Japanese oak wilt disease and surrounding forest compositions, Mendeley Data, v1; 2015.

ttps://doi.org/10.17632/xwj98nb39r.1.

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PARECER CONSUBSTANCIADO DO CEP

DADOS DO PROJETO DE PESQUISA

Título da Pesquisa: MÉTODOS DE ENVELHECIMENTO IN VITRO NA RESISTÊNCIA DE UNIÃO AO TESTE DE MICROCISALHAMENTO DE SISTEMAS ADESIVOS

Pesquisador: ALEXANDRE HENRIQUE SUSIN

Área Temática:

Versão: 1

CAAE: 67687417.9.0000.5346

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

Patrocinador Principal: Financiamento Próprio

DADOS DO PARECER

Número do Parecer: 2.054.447

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Continuação do Parecer: 2.054.447

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Situação do Parecer:
Aprovado

Necessita Apreciação da CONEP:
Não

SANTA MARIA, 09 de Maio de 2017

Assinado por:
CLAUDEMIR DE QUADROS
(Coordenador)