Ana Laura Rezende Vilela

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Evaluation of factors that interfere on the analysis of methodology and longevity of adhesive restorations

Tese apresentada à Faculdade de Odontologia da Universidade Federal de Uberlândia para obtenção do Título de Doutora em Odontologia na Área de Concentração em Clínica Odontológica Integrada

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Orientador: Prof. Dr. Murilo de Sousa Menezes

Banca Examinadora:
Prof. Dr. Murilo de Sousa Menezes
Prof. Dr. Paulo César de Freitas Santos-Filho
Profª Drª. Karla Zancopé
Profª Drª Fernanda Pereira Silva
Prof. Dr. Crisnicaw Verissimo.

Uberlândia 2021

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V699
2021

Vilela, Ana Laura Rezende, 1990-
Avaliação de fatores que interferem na metodologia de análise e na longevidade de restaurações adesivas [recurso eletrônico] / Ana Laura Rezende Vilela. - 2021.

Orientador: Murilo de Sousa Menezes .
Tese (Doutorado) - Universidade Federal de Uberlândia, Pós-graduação em Odontologia.
Modo de acesso: Internet.
Disponível em: http://doi.org/10.14393/ufu.te.2021.397
Inclui bibliografia.

1. Odontologia. I. , Murilo de Sousa Menezes,1979-, (Orient.). II. Universidade Federal de Uberlândia. Pós-graduação em Odontologia. III. Título.

CDU: 616.314

Bibliotecários responsáveis pela estrutura de acordo com o AACR2:

Gizele Cristine Nunes do Couto - CRB6/2091

UNIVERSIDADE FEDERAL DE UBERLÂNDIA
Coordenação do Programa de Pós-Graduação em Odontologia
Av. Pará, 1720, Bloco 4L, Anexo B, Sala 35 - Bairro Umuarama, Uberlândia-MG, CEP 38400-902
Telefone: (34) 3225-8115/8108 - www.ppgoufu.com - copod@umuarama.ufu.br

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Programa de Pós-Graduação em:	Odontologia				
Defesa de:	Tese Doutorado, número 67, PPGODONTO				
Data:	Trinta de Julho de Dois Mil e Vinte e Um	Hora de início:	08:00	Hora de encerramento:	[12:52]
Matrícula do Discente:	11713ODO001				
Nome do Discente:	Ana Laura Rezende Vilela				
Título do Trabalho:	Avaliação de fatores que podem interferir na metodologia de análise e na longevidade de restaurações adesivas				
Área de concentração:	Clínica Odontológica Integrada				
Linha de pesquisa:	Propriedades Físicas e Biológicas dos materiais Odontológicos e das estruturas dentais				
Projeto de Pesquisa de vinculação:	Propriedades Físicas e Biológicas dos materiais Odontológicos e das estruturas dentais				

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Referência: Processo nº 23117.048213/2021-43 SEI nº 2921158

DEDICATÓRIA

À Deus, por estar sempre em minha vida e me guiando desde o início dessa jornada. Por ter me dado saúde, sabedoria, conhecimento e força de vontade para buscar o conhecimento.

Aos **meus pais**, **Jose Manoel e Soraya** que sempre me apoiaram em cada decisão, nunca mediram esforços para me proporcionar o melhor, obrigada pelo amor incondicional. Sem o amparo de vocês eu jamais teria conseguido vencer mais essa etapa. Amo vocês!

A **minha irmã Letícia**, pelo companheirismo e por sempre estar disposta a me ajudar. Obrigada pela amizade e paciência. Amo você.

Ao **Alexandre**, pelo companheirismo, atenção e sábios conselhos. Essa tese e toda a minha caminhada profissional não teria o mesmo êxito sem suas contribuições. Obrigada!

Aos meus avós, tios e primos e amigos, pelo carinho e orações.

AGRADECIMENTOS

Ao **professor Dr. Murilo de Sousa Menezes**, muito obrigada por todas as oportunidades e principalmente pela confiança depositada em mim. Agradeço pela paciência e pelo compartilhamento de conhecimentos. Admiro-te por ser um profissional com amplo conhecimento e ideias inovadoras, mas sempre com humildade perante o próximo.

A **Fernanda, Marcela, Giselle, Thaís, Lucas, Thamires e Renata** que são amigos para todas as horas, que sempre estão comigo em todos os momentos, sempre me apoiando e aconselhando. Obrigada pela força que foi essencial para superar todos os obstáculos e seguir em frente.

Aos alunos de iniciação científica: **Pedro, Amanda e Grazielly** pelo apoio e dedicação durante a realização dos trabalhos.

Aos **amigos da pós-graduação**: Rafael, Lívia, Renata, Daniela, Lucas, Caio e Júlia, Karla e Thais, Marcela e Letícia, Guilherme, Juliana e Camila, amigos que a pós-graduação me deu e vou levar para vida toda. Agradeço pela troca de experiências. Todos vocês foram muito importantes para o meu crescimento pessoal e profissional.

A **minha segunda família:** Maria Inês, Laerson, Ivan, Douglas, Lilian e Maria. Obrigada pela atenção e carinho, por nunca medirem esforços para me ajudar e amparar quando estava longe da minha família.

Aos **professores e funcionários da ESTES/UFU**, em especial: Samara, Aline, Ludmila, Juliana, Bruno e Sirlete pela ótima experiência como professora substituta.

Aos demais **professores da área de Dentística e Materais Odontológicos,** Carlos José Soares, Paulo Vinicius Soares, Paulo Sérgio Quagliatto, Paulo César Santos-Filho, Veridiana Resende Novais, Gisele Rodrigues Silva e Roberto Elias Campos, pela convivência e ensinamentos. Vocês são exemplos de dedicação e vocação para a docência.

A todos os **professores de pós-graduação**, muito obrigada pelos conhecimentos transmitidos durante todo a pós graduação.

Aos **colegas de doutorado**, que tornaram essa jornada muito prazerosa.

Aos **funcionários da FOUFU, em especial aos da Pós-graduação, aqui representados pela** Graça, Brenda, Laís, John e Bruno. Obrigada por realizarem seus trabalhos com tanta dedicação e paciência, facilitando nossas tarefas diárias.

Ao **Programa de Pós-graduação da Faculdade de Odontologia da Universidade Federal de Uberlândia**, pela minha formação profissional. Agradeço a ótima equipe de profissionais que fazem parte da história dessa instituição e que se tornaram parte também da minha história.

Aos **professores** André, Paulo Vinícius, Raposo da odontologia e a Letícia e professor Washington da engenharia mecânica, pela dedicação e contribuição aos trabalhos.

A **LAMEV-FEQui/UFU e Engenharia Mecânica UFU**, obrigada pela oportunidade de enriquecer meu trabalho, através dos equipamentos disponibilizados por vocês.

A **CAPES** pelo apoio e incentivo a pesquisa e ensino.

Ao **CPbio**, obrigada pela disponibilidade de equipamentos e materiais que me permitiram realizar a minha pesquisa.

SUMÁRIO

Resumo

RESUMO

Vários fatores podem influenciar na longevidade das restaurações: protocolo restaurador, material, bem como os cuidados do paciente com a restauração. O objetivo geral desse trabalho foi avaliar os fatores que podem interferir na metodologia de análise e na longevidade das restaurações adesivas. Este estudo foi dividido em 4 capítulos de acordo com cada objetivo específico: **Capitulo 1)** Avaliar a influência do intervalo de tempo, entre os desafios corrosivos e abrasivos (imediato, 15 minutos e 30 minutos), na rugosidade de superfície e na dureza de uma resina composta nanoparticulada; **Capitulo 2)** Analisar a influência de diferentes parâmetros na metodologia de microcisalhamento, como espessura do fio ortodôntico (0,2 mm e 0,7mm) e a distância entre os corpos de prova (1,5 e 3mm), na resistência de união e distribuição de tensões de uma cerâmica de dissilicato de lítio; **Capitulo 3)** Avaliar a eficácia de diferentes silanos (pré-hidrolisados e de hidrólise imediata), na forma de armazenamento (em temperatura ambiente ou em 5ºC) e na resistência de união de reparo em resina composta e **Capitulo 4)** Influência da estabilidade hidrolítica na resistência de união de silanos e de um adesivo universal em reparos de resina composta. Os métodos experimentais utilizados foram: rugosidade (capítulo 1); microdureza (capítulo 1); microscopia eletrônica de varredura (capítulo 1, 2, e 3); microcisalhamento (capítulo 2,3 e 4); análise por elementos finitos (capítulo 2); ângulo de contato (capitulo 3). Os resultados encontrados mostram que: **1)** A imersão em bebida ácida seguida imediatamente pela escovação com pasta clareadora, aumentou a rugosidade de superfície da resina composta; o intervalo de 30 minutos entre a ingestão de bebidas ácidas e a escovação demonstrou importante para reduzir os efeitos deletérios nas restaurações de resina composta; e a microdureza não foi influenciada pelo intervalo de ingestão de bebidas ácidas e a escovação; **2)** O diâmetro do fio ortodôntico influenciou na resistência de união ao microcisalhamento, sendo que o fio com diâmetro de 0,2mm apresentou melhor valores de adesão e as distâncias entre os corpos de prova (1,5 ou 3mm), não influenciaram na resistência de união e na concentração de tensões dos materiais utilizados**; 3)** O silano e a forma de armazenamento, após abertura, influenciaram na resistência

de união em reparos em resina composta. O silano pré-hidrolisado apresentou maior resistência de união inicialmente e o silano de hidrólise imediata manteve os valores de adesão mesmo após um ano de armazenamento em temperatura ambiente, quando comparado ao armazenamento a 5ºC. **4)** A estabilidade hidrolítica do silano foi pior para o silano pré-hidrolisado. Após o processo de envelhecimento o adesivo universal apresentou maiores valores de adesão quando comparado com os silanos pré-hidrolisados ou de hidrólise imediata. Pode-se concluir que a escovação e a ingestão de bebidas ácidas podem influenciar na rugosidade da resina composta nanoparticulada e que o silano pré-hidrolisado é um produto instável, independente do frasco ser envelhecido lacrado ou após já ter sido utilizado e que o método de produção das amostras para o teste de microcisalhamento pode interferir nos resultados finais.

PALAVRAS-CHAVE: resina composta; silano; microcisalhamento

Abstract

ABSTRACT

Several factors can influence the longevity of restoration: restorative protocol, material, as well as patient care with the restoration. The general objective of this study was to evaluate the factors that can interfere in the analysis methodology and in the longevity of adhesive restorations. Thus, this study was divided into 4 chapters according to each specific objective: **Chapter 1)** Evaluate the influence of the time interval, between corrosive and abrasive challenges (immediate, 15 minutes and 30 minutes), on surface roughness and hardness of a nanoparticulate composite resin**; Chapter 2)** Analyze the influence of different parameters on the microshear methodology, such as the thickness of the orthodontic wire (0.2 mm and 0.7 mm) and the distance between the specimens (1.5 and 3 mm), on the bond strength and stress distribution of a lithium disilicate ceramic; **Chapter 3)** Evaluate the effectiveness of different silanes (pre-hydrolyzed and immediate hydrolysis), in the form of storage (at room temperature or at 5ºC) and in the bond strength of composite resin repair and **Chapter 4)** Influence of hydrolytic stability on the bond strength of silanes and a universal adhesive in composite resin repairs. The experimental methods used were: roughness (chapters 1); microhardness (chapter 1); scanning electron microscopy (chapters 1, 2, and 3); microshear (chapters 2, 3 and 4); finite element analysis (chapter 2); contact angle (chapter 3). The results showed that: **1)** Immersion in an acid beverage, immediately followed by brushing with bleaching paste, increased the surface roughness of the composite resin; the 30-minute interval between drinking acidic beverage and brushing was shown to be important to reduce the deleterious effects of composite resin restorations; and microhardness was not influenced by the interval between drinking acidic beverage and brushing; **2)** The diameter of the orthodontic wire influenced the microshear bond strength, and the wire with a diameter of 0.2mm had better adhesion values and the distances between the specimens (1.5 or 3mm) did not influence the strength bonding and stress concentration of the materials used; **3)** The silane and the storage form, after opening, influenced the bond strength in composite resin repairs. The pre-hydrolyzed silane initially presented higher bond strength and the immediate hydrolysis silane maintained adhesion values after

one year of storage at room temperature, when compared to storage at 5ºC. **4)** The hydrolytic stability of the silane was worse for the pre-hydrolyzed silane. After the aging process, the universal adhesive showed higher adhesion values when compared to pre-hydrolyzed or immediate hydrolysis silanes. It can be concluded that brushing and the ingestion of acidic beverages can influence the roughness of the nanoparticulate composite resin and that the pre-hydrolyzed silane is an unstable product, regardless of aged with the sealed bottle or after it has already been used and that the method of production of samples for microshear testing may interfere with the final results

KEYWORDS: Composite resin; silane; microshear.

Introdução e referencial teórico

1. INTRODUÇÃO E REFERENCIAL TEÓRICO:

As restaurações adesivas são muito utilizadas na odontologia restauradora, por apresentarem inúmeras vantagens quando comparadas aos demais materiais restauradores, pois possibilitam: maior conservação de estrutura dentária sadia (1), estética satisfatória (1), adesão a estrutura dentária e (2) boa longevidade (3).

Restaurações com estes materiais se aderem à estrutura dentária por união química ou micromecânica (1, 2, 4). A adesão química é caracterizada pela interação do material com a estrutura dentária ou entre materiais restauradores (2, 5). Já a adesão micromecânica, se dá por meio da formação de retenções micromecânicas na superfície do dente, seja ela com a infiltração dos monômeros nas microporosidades geradas na superfície do esmalte, ou entre as fibrilas colágenas, após a remoção da smear layer na dentina (1, 2, 4).

Ao longo dos anos, muitos estudos avaliaram a longevidade clínica das restaurações anteriores e posteriores (3, 6-8). Enquanto os principais motivos para o insucesso em restaurações em resina composta de dentes posteriores são cárie e fratura do dente e/ou restauração, em dentes anteriores o fator estético tem um papel fundamental na decisão de nova intervenção por parte do cirurgião dentista e do paciente (3, 6-8). A decisão após avaliar clinicamente uma restauração mais antiga pode variar entre não intervir e somente acompanhar, reparar ou substituir (9). A decisão selecionada determinará a menor ou maior longevidade da restauração (9). Várias razões podem influenciar na longevidade das restaurações, como fatores relacionados ao risco do paciente, como o risco de cárie dentária, hábitos de higienização e parafuncionais, idade, fatores socioeconômicos e alimentação; fatores dependentes do operador como experiência do operador (10), técnica de restauradora (12) e de fotoativação (11); fratura dentária e estética da restauração (3, 13, 14).

Estes critérios que interferem na longevidade das restaurações ou na necessidade de troca destas, foram elaborados por CVAR e Ryge em 1964 (15)

e atualizada por Bayne e Schmalz em 2005 (16). Os critérios foram denominados de “US Public Health Service- (USPHS), que envolvem as seguintes alterações: mudança de cor, manchamento da margem cavo-superficial, forma anatômica irregular, adaptação marginal e presença de da doença cárie. Em 2007 e 2008, surgiu mais um grupo de critérios clínicos para restaurações diretas e indiretas denominada de FDI- World Dental Federation. Os critérios foram classificados em três grupos: estético, funcional e biológico (17, 18). Estes critérios foram atualizados por Hickel e colaboradores em 2010. Entre os critérios estéticos estão: polimento da superfície, manchamento, alteração de cor e translucidez, e forma anatômica (19).

A alteração destes fatores estéticos definidos por Hickel 2010, estão relacionados a vários fatores entre eles: o tipo de alimentação e a técnica de escovação. A alimentação influência na longevidade das restaurações diretas por meio da pigmentação e acidez do alimento (20, 21). Os pigmentos de açaí, vinho e outros alimentos, penetram nos materiais resinosos gerando alterações de cores que podem resultar na necessidade de substituição, reparo ou apenas realização de novo acabamento e polimento das restaurações (22, 23). A acidez influência devido ao baixo pH que pode gerar uma degradação da matriz resinosa e conseguinte remoção de partículas de carga, resultando maior rugosidade da superfície deste material (21, 24-26). A degradação ácida começa com a absorção de água que se difunde internamente através das interfaces das partículas de carga da matriz, poros e outros defeitos do material resinoso, acelerada por um baixo pH que causa o descolamento das partículas de carga ou mesmo degradação hidrolítica da interface das partículas (26, 27), resultando asssim em menor dureza e maior rugosidade na superfície das resinas compostas (26). Além da acidez o teor alcoólico também influencia para maior absorção de pigmentos (22, 24, 28). O álcool facilita a penetração dos fluidos e pigmentos para o interior da resina composta (22).

A escovação pode influenciar na rugosidade e manchamento da restauração a depender da abrasividade dos dentifrícios utilizados. A abrasividade dos dentifrícios é medida pelo RDA (Relative dentin abrasivity) (29). Os abrasivos presentes nos dentifrícios são importantes para evitar a

pigmentação da superfície do dente (29, 30). No entanto, quanto maior a abrasividade do dentifrício maior a chance de gerar rugosidade na superfície da restauração (24, 31, 32) ou do dente (24), o que potencializa a possibilidade de descoloração deste. As pastas clareadoras geralmente são as mais abrasivas do mercado (24, 33, 34). Vale salientar que a escovação, quando realizada com técnica e dentifrícios adequados, resulta em menor retenção de placa bacteriana e pigmentação das restaurações em resinas compostas (35).

Demarco 2015 (3) relatou em seu artigo que o índice de falha anual das restaurações em resina composta em dentes anteriores é de 0-4.1%, sendo a perda da estética a causa principal de falha destas restaurações. Muitas vezes as falhas geradas nas restaurações adesivas, principalmente de resina composta, podem ser reparadas e não substituídas (8, 36, 37). O reparo é um procedimento em que se troca apenas a porção da restauração que apresenta a falha, mantendo parte da restauração antiga na cavidade oral. Estudos mostram que o reparo de restaurações prolonga a sobrevida das restaurações (8, 9, 36). O reparo das restaurações pode ser realizado nas seguintes situações: correções de *gaps* marginais, manchamento marginal localizada, reparo de fraturas que não causaram prejuízos aos esmalte e a dentina adjacentes, lascamento da margem da restauração, desgaste e correção da estética (38). As técnicas minimamente invasivas reduzem o desgaste da estrutura dentária sadia, o risco de danos iatrogênicos, bem como o custo do tratamento (37). O reparo aumenta a vida útil das restaurações de 65,92% para 74,61%, em 10 anos (39). Porém, podemos perceber que após o reparo, a parte remanescente da restauração original, apresenta taxa de sucesso maior do que a parte reparada (9). Uma possível explicação para esta maior taxa de sucesso do remanescente original da restauração, pode estar relacionada a falhas de adesão pois, embora uma série de protocolos de reparo tenham sido publicados nos últimos tempos (9), a adesão de uma restauração antiga a uma nova restauração, continua sendo um desafio para a odontologia (9).

O sucesso dos procedimentos de reparo requer uma adesão durável entre a restauração antiga e a nova (40). Os novos incrementos de resinas compostas utilizados nos reparos se aderem as resinas compostas das restaurações de

duas formas: por meio de embricamento mecânico ou pela adesão química com as partículas de carga e a matriz orgânica (40, 41). O protocolo de reparo é um procedimento complexo e necessita ser realizado de forma criteriosa e detalhada para que a restauração tenha maior longevidade (40).

Ao se realizar um reparo, as resinas compostas recentemente polimerizadas são mais reativas do que as resinas compostas das restaurações já realizadas a algum tempo, devido à redução de monômeros livres presentes na superfície das restaurações pré-existentes, devido a absorção de água e a plastificação, que são responsáveis por relaxar as ligações físicas entre as cadeias poliméricas, gerando a lixiviação de monômeros livres (40, 42). Por isso, é necessário a realização de tratamento de superfície destas resinas pré-existentes, podendendo estes serem tratamentos físicos e químicos (40). Entre os tratamentos físicos utilizados temos: a asperização com pontas diamantadas finas, jateamento com oxido de alumino e o uso do plasma de Argônio (37, 40), que resultarão em rugosidades e consequentemente aumento da resistência mecânica de união (43). A asperização por jateamento cria microretenções e superfície uniformemente rugosa, o que amplia a área de contato superficial que vai interagir com o agente de união e o novo incremento de compósito resinoso utilizado no reparo (40, 44). O condicionamento com ácido fosfórico, independente da concentração utilizada, não resulta em efeitos diretos nas características da superfície do compósitos, cerâmicas ou metais. No entanto, o condicionamento ácido apresenta efeito benéfico nas taxas de retenção do reparo, atribuido ao efeito de limpeza e desengorduramento dessas superfícies (37).

A adesão química é realizada principalmente pelos agentes de união silânicos. Os silanos são produtos bifuncionais que se ligam a parte inorgânica (sílicas) e com a matriz orgânica (monômeros resinosos) (45). A união com a parte inorgânica ocorre por meio da formação de ligações de siloxanos, que são ligações químicas covalentes com as partículas de carga (46) e realizam uma co-polimerização com a matriz resinosa (40). A utilização do silano em reparo de restaurações de resina composta, aumenta a resistência de união entre a resina composta da restauração antiga e o novo incremento utilizado para o reparo,

principalmente quando associado a aplicação de uma camada de sistema adesivo, uma vez que a resina composta da restauração possui baixa quantidade de monômeros reativos (40, 41, 44-46). Existem vários tipos de produtos que contém silano, que podem estar na sua forma hidrolisada ou não hidrolisada. Os silanos hidrolisados são os tipos de silanos mais utilizados na prática odontológica, porém, este produto apresenta uma meia vida curta, sendo que sua efetividade vai sendo perdida quando o produto é aberto para ser utilizado pela primeira vez (45). Como mencionado acima, a última etapa para o tratamento de superfície de restaurações em resina composta a serem reparadas, é a aplicação de uma camada de sistema adesivo (47). O adesivo tem a função de penetrar nas irregularidades da resina composta antiga e aumentar a molhabilidade do compósito resinoso (37). Um dos sistemas adesivos mais utilizados neste procedimento é o Universal, que apresenta em algumas formulações silano, que pode substituir a aplicação do silano em alguns casos (2).

Objetivo Geral

2. Objetivo Geral:

O objetivo geral deste trabalho foi avaliar os fatores que interferem na metodologia de análise de resistência de união por microcisalhamento e a longevidade das restaurações por meio da análise da topografia de superfície das resinas compostas após desafios corrosivos e abrasivos e por meio da análise da efetividade dos silanos utilizados em reparo em resina composta.

Objetivos Específicos

3. Objetivos Específicos:

3.1. Objetivo específico 1: O objetivo deste estudo foi avaliar o efeito do intervalo entre o desafio corrosivo e abrasivo na rugosidade e dureza de uma resina composta nanopartículada. Por meio da análise da rugosidade de superfície, microdureza Knoop e Mev.

3.2. Objetivo específico 2: O objetivo deste trabalho foi avaliar os diferentes parâmetros no teste de μSBS como a distância entre os espécimes e a espessura do fio ortodôntico na resistência de união e na distribuição de tensão. Por meio da análise de microcisalhamento e análise por elementos finitos.

3.3. Objetivo Específico 3: O objetivo deste trabalho foi analisar a eficácia de diferentes tipos de silanos armazenados em temperatura ambiente ou em 5ºC na resistência de união de reparos em resina composta. Por meio dos testes de microcisalhamento e ângulo de contato.

3.4. Objetivo Específico 4: O objetivo deste trabalho foi avaliar a resistência de união e a estabilidade hidrolítica de 2 tipos de silanos e um adesivo universal em reparos de resina composta utilizando um frasco fechado novo e outro envelhecido.

Capítulos

Essa tese foi dividida em 4 capítulos:

4.1. Capítulo 1 - Artigo aceito para publicação no periódico Europian Journal of Dentistry: Effect of interval time between corrosive and abrasive challenges on a nanoparticulate composite resin.

4.2. Capítulo 2- Artigo em revisão para publicação no periódico Journal of Adhesive Dentistry: Influence of different microshear parameters on bond strength and stress distribution.

4.3. Capítulo 3- Artigo nas normas para submissão para publicação no periódico Brazilian Dental Journal- Influence of silane storage time after opening on bond strength and contact angle of composite resin repair.

4.4. Capítulo 4- Artigo nas normas para submissão para publicação no periódico Brazilian Oral Research- Influence of silane, universal adhesive and the aging of the closed bottle on the repair bond strength in composite resin.

Capítulo I

Effect of interval time between corrosive and abrasive challenges on a nanoparticulate composite resin

Ana Laura Rezende Vilela[1], Alexandre Coelho Machado[2], Lucas Lemes Queiroz[3], Pedro Henrique Mauro Batista[4], André Luís Faria-e-Silva [5], Murilo de Sousa Menezes [6]

1. Ana Laura Rezende Vilela. DDS, MSc. Dental School, Federal University of Uberlândia, Brazil- analaurarvilela@ufu.br
2. Alexandre Coelho Machado. DDS, MSc, PhD. Professor, Technical Health School, Federal University of Uberlândia., Brazil- alexandrecoelhomachado@ufu.br
3. Lucas Lemes Queiroz. DDS, MSc. Dental School, Federal University of Uberlândia, Brazil -lucaslemesq@gmail.com
4. Pedro Henrique Mauro Batista. DDS, Graduate student, Dental School, Federal University of Uberlândia, Brazil. - phmaurobatista@gmail.com
5. André Luís Faria-e-Silva. DDS, MSc, PhD, Professor, Department of Restorative Dentistry, Dental School, Federal University of Sergipe, Brazil - fariaesilva.andre@gmail.com
6. Murilo de Sousa Menezes. DDS, MSc, PhD, Professor, Department of Restorative Dentistry and Dental Materials, Dental School, Federal University of Uberlândia, Brazil - murilomenezes@ufu.br

Number of words: 3.497
Number of figure: 2
Video Files: 0

Corresponding author:

Prof. Dr. Murilo de Sousa Menezes

Federal University of Uberlandia, Dental School, Department of Restorative Dentistry and Dental Materials. Avenida Pará, 1720, Bloco 4L, Anexo A, Sala 34, Campus

Umuarama Uberlândia - Minas Gerais – Brazil, Tel.: +55 34 3225-8105; Fax.: +55 34 3218 2279 E-mail address: murilomenezes@ufu.br

Abstract

Objective: Evaluate the effect of interval time after acidic beverage intake and brushing on roughness and hardness of resin composite.

Materials and Methods: Nanofilled resin composites were tested according interval time (No-interval, 15 or 30 min) between aging media (Isotonic- sports drink) and brushing. Specimens (n=9) were subjected to three cycles daily for 5 days with immersion in beverage followed by simulated brushing (585 strokes). The brushing (control) group was submitted only in brushing cycles. Roughness and Microhardness were analyzed in the baseline and end of the experiment. Surface morphology was analyzed using Scanning electron microscopy (SEM).

Statistical analysis: Data were analyzed by One-way ANOVA and Tukey's HSD (α=0.05).

Results: Roughness was higher in no-interval group and lower in 30 min and control. The 15 min present no statistical difference between control, 30 min and no-interval. The hardness not present difference between groups. The SEM showed the no-interval more roughness than 15 min, 30 min, control and baseline.

Conclusion: The interval time between erosive and abrasive challenge is important to preserve the smoothness surface of composite resin.

Keywords: composite resins, toothbrushing, beverages.

Introduction

The composite resin restorations are widely indicated for treatment of caries,[1] esthetic reasons and dental wear management.[1] The frequent use of composite resins are related to greater preservation of the dental structure, low cost, simple technique and lower clinical time.[2] This material consists of an organic matrix, filler inorganic particles and silane bonding agent.[3] The size, shape and amount of the filler particles improve mechanic properties and polishing.[2] Currently, the smallest scale of particles used are the nano fillers and the composite resins with exclusivity of nanometric particles are classified as nanofilled. Moreover, the failure rates (AFR) of composite resin restorations reaches 4.1% and 2.2 % for anterior and posterior teeth.[3]

The wide range of clinical applications of composite resin is assessed mainly through their ability to mimic the optical effects of dental structures, mechanical, adhesion to dental structures and physical properties.[2] Despite all these advantages, the resin composite restorations are susceptible to surface changes due to aging caused by acidic abrasive substances. These aging can affect the aesthetic properties and also the smoothness and strength of the restoration, becoming unsatisfactory over the time. According to World Dental Federation (FDI), the failures criteria for direct restoration are surface lustre, staining, color match, translucency and esthetic anatomical form [4]. The main causes for these undesirable effects can be assigned to insufficient polymerization, unsatisfactory finishing and polishing, parafunctional habits, operator experience and incorrect and unsatisfactory oral hygiene.[5]

The roughness or smoothness surface are related to polishing technique. A deficiency in the finishing, inadequate polishing procedure, acidic diet and deleterious

brushing habits increase the roughness surface, over the time, resulting in aesthetic damage to the restoration.[2] Moreover, a rougher surface increase the potential biofilm accumulation,[6] increasing the gingival inflammation and maintaining color stability for less time.[2] The oral hygiene is important to teeth and periodontal healthy, through of removing dental plaque and superficial staining on teeth and composite resin restoration.[5] The abrasives present in toothpaste have function of contribute to biofilm remove and reduce restorations and teeth pigmentation after eating food and drinks.[7] However, toothbrush and toothpaste can negatively affect the smoothness surface of composite resin according to the abrasiveness of the dentifrice, stiffness of toothbrush bristles, major associated with acidic diet.[8]

The consumption of sports and energy drinks is currently increasing, mainly due to the population's greater concern about health and body aesthetics [9]. Moreover, beverages present low pH (3.8-2.3) generates dental corrosion[10] and cause degradation of the composite resin organic matrix, increasing the surface roughness and decreasing hardness and flexural strength.[11] The consumption of acidic beverages and oral hygiene is a daily practice of the majority of the world population. The other factor that may be associated with the wear of composite resins is through brushing with high abrasive dentifrice, as whitening toothpastes.[12]

Therefore, the objective of this article is to evaluate the influence of interval time between corrosive and abrasive challenge on surface roughness and hardness of nanofilled composite resins. The null hypothesis of the study is the interval time between acidic beverage and brushing will not affect the surface roughness and hardness of composite resins.

Materials and Methods

Experimental design

This was a laboratory study conducted to evaluate the independent variable 'time elapsed between corrosive and abrasive challenges' on changes in roughness and hardness (dependent variables). Four levels of independent variable were defined based on interval time between the challenges (no-interval, 15 min, or 30 min) and absence of corrosive challenge (control) – Figure 1.

Specimen preparation

Thirty-six disc-shaped specimens of the nano-filled composite resin Filtek Z350 X (3M ESPE, St. Paul, MN, USA), shade A1, were built-up using a cylindrical teflon mold (6 mm of diameter, and 1 mm of thickness). After the composite insertion, the mold was covered with mylar strips, and the material maintained under digital pressure for 10 s before the light-activation. The specimens were light cured for 20 s with the light-emitting diode (LED) Bluephase N (Ivoclar Vivadent, Schaan, Liechtenstein; irradiance ≈ 1.000 mW/cm²). The polymerized specimens were stored in distilled water at 37ºC for 24 h before the finishing procedures with SiC sandpapers (# 600, 800, 1.200, and 2.000) under water irrigation (Politriz Universal, Arotec, São Paulo, SP, Brazil). Following, the specimens were cleaned in an ultrasonic bath with distilled water for 10 min and stored in artificial saliva at 37 ºC.

Measurements at baseline

The average surface roughness (Ra) of the specimens was assessed using a surface roughness tester (Surftest SJ- 410; Mitutoyo Corp, Tokyo, Japan) at a constant speed of

0.25 mm/s and cut-off of 0.8 mm. Three readings were carried out for each specimen modifying direction at approximately 120° between two consecutive readings. The mean surface roughness of the three readings was recorded for each specimen. Moreover, the knoop microhardness (KHN) of specimens was determined using a microhardness tester (FM-7000, Future-Tech Corp, Kawasaki, Japan) with a diamond Knoop indenter. Five equidistant indentations were carried-out with a load of 0.98 N for 15 s on the specimen's surface, and averaged hardness was recorded for each specimen.

Corrosive challenge

Twenty-seven specimens were immersed for 5 min in 5 ml of lemon flavor isotonic beverages (PowerADE Lemon, Coca-Cola, Atlanta, GA, USA) with agitation of 120 rpm,[11] three times a day for 5 days. The pH of beverages (≈ 3.8) was measured daily. The nine remaining specimens were used as a control. Following, the specimens underwent to the corrosive challenge were kept in artificial saliva for either 15 or 30 min before the abrasive challenge; or abraded under toothbrushing movements without any interval (n = 9).

Abrasive challenge

All specimens were brushed using soft toothbrushes (Colgate Classic, Colgate-Palmolive, São Bernardo do Campo, SP, Brazil) attached to a toothbrushing simulator machine (Odeme Biotechnology, Joaçaba, SC, Brazil). A toothpaste solution was prepared using the toothpaste Colgate Iluminous White (Colgate-Palmolive, São Bernardo do Campo, SP, Brazil) and distilled water at 1:2 ratios by weight.[17,18] Specimens were underwent to 585 brushing cycles under a constant load of 200g, three times a day for five days. Each cycle was determined by the one back and forth movement of the

brush. After the end of the abrasive challenge, the roughness and hardness of specimens were assessed again as described before.

Qualitative analysis of surface

Three specimens of each experimental condition were randomly analyzed using a scanning electron microscope (Leica EM SCD50, Leica Microsystems, Wetzlar, Lahn-Dill, Germany). Specimens were sputter-coating with a thin film of gold at 15.0 Kv. The images were taken with 1.000x magnification selecting a more representative area in the specimen.

Data analysis

Changes on roughness and hardness were calculated by subtracting the values measured after corrosive and/or abrasive challenges from those observed at baseline. Normal distribution of data and homogeneity of variance were assessed by Shapiro-Wilks and Levene`s test, respectively. Data were analyzed by one-way ANOVA and post-hoc Tukey`s test. A confidence level of 95% was pre-set for all data analysis.

Results

The results for changes in roughness and hardness are summarized in Table 1. One-way ANOVA showed that the treatment affected the changes on roughness of specimens ($p = .005$). The no-interval specimens presented the highest values of change on roughness (11.7 [14.4]), but without statistical difference for elapsing 15 min (-3.74 [11.8]) between the challenges. There was no statistical difference between the intervals of 15 min and 30 min (-5.81 [11.7]), and the control (-9.4 [10.9]). One-way ANOVA showed that the treatment did not affect the changes on surface hardness ($p = .858$) for

control (-6.1 [14.3]), no-interval (-9.9 [14.3]), 15min (-7.2 [9.2]) and 30min (-10.3 [13.0]).

The smoothest surface was observed at baseline (Figure 2A). The control group (Figure 2B) presented a smoother surface (no groove neither exposed fillers) than specimens underwent to corrosive challenge. No-interval between the challenges (Figure 2C) yielded the deepest grooves and most pronounced irregularities on composite surface. Slight grooves and few irregularities were observed when an interval of 15 (Figure 2D) or 30 min (Figure 2E) were elapsed between the challenges.

Discussion

The acid beverage intake associated with brushing in sequence can generate changes on tooth surface[10] and on surface of composite resin restoration, which was confirmed with present study. Therefore, the null hypothesis was rejected because the interval time between corrosive and abrasive challenges influence the surface roughness of nanofilled composite resins.

The worldwide consumption of soft drinks, fruit juice, sports drinks and energy drinks has increased in recent decades.[13] The consummation of some acidic beverages is associated with the healthy life style and to the improvement of the performance in physical exercises.[14] The isotonic beverage is an acidic beverage widely used by athlete's aid the body maintain proper hydration and supplement minerals that are lost in sweat during excessive exercising.[14] Despite these indications, the isotonic present a low pH (3.8) and it is associated with the capacity of dental wear and restorative materials.[15] When the teeth are exposed to acid substances and with a low concentration of Ca^{2+}, PO_4^{3-}

and OH^-, there is a tendency for enamel to release more of these ions, and the demineralization process is more intense.[13] During the oral intake, the acid beverage is swished in the mouth and result in higher contact of the beverage with the tooth and restorative surfaces.[15] The agitation of the acid fluid in the oral cavity promotes the continuous ions out-flow from the enamel and will lead to a more intensive corrosive-erosive process.[10, 13]

In addition, the lifestyles and the association with other factors, such as parafunctional habits and occlusal factors generate dental wear at levels that require restorations to replace the loss of tooth structure. The most common approach for restoring severely worn teeth was the use of resin-based composite.[16] The survival rate of composite resin restorations on worn teeth is 85% in 7 years.[16] This restoration must survive in an environment that the teeth did not could resist, so, it is essential that the patient changes their habits and perform all the precautions and care necessaries to maintain the quality of the restorations.[9, 12, 16]

The immediate group showed an increase in surface roughness after five days of challenges. The isotonic is an aggressive immersion media due to the presence of citric acid in its composition and generates a chemical degradation of the organic matrix with, consequently, increase of the surface roughness.[12] Furthermore, the chemical degradation, induced by low pH solution, increases the damage promoted by tooth brushing.[17] This was confirmed on the roughness surface values and SEM, which showed that brushing immediately after immersion in beverages with acidic pH increase the composite resin roughness. The immediate group showed higher values of roughness surface after the corrosive and abrasive challenges than others groups, confirming the greater degradation of the resin matrix.

The 30 min group did not present statistically change in surface roughness than control. The immersion in artificial saliva after the corrosive challenge generated a neutralization of the acid environment, reducing the potential damage of the brushing and organic matrix degradation [11]. In other hand, the 15 min group present similar roughness with all groups. The similarity between the 15min and the immediate group is due to the incomplete pH neutralization. However, the SEM images showed a reduced inorganic particle extruded in composite resin surface on 15 and 30 min, which explain the similarity between control, 15 and 30min.

The toothpastes are used for oral hygiene but, usually, these toothpastes present secondary function, such as whitening.[18] Many patients desire a smile with white teeth, however, instead they looking for a professional bleaching they choose for others alternatives, as whiting toothpaste. The International Organization for Standardization (ISO) recommends that the relative dentin abrasiveness (RDA) of a toothpaste does not exceed 250.[19] The toothpaste abrasiveness is important to prevent extrinsic staining of teeth and composite resin restoration [7]. Whitening toothpaste present higher RDA (175) than other toothpaste.[19] Moreover, the toothpaste composition must be considered beyond the RDA, because the ability to remove the extrinsic stains is also related to the chemical composition.[20]

The potential wear of composite resin is material dependent. Different composite resins are different influenced by abrasives pastes,[21] although the filler size of the composite resin is not determinant in the degradation process.[18] This result can be explained due to no difference among the whiting and conventional toothpaste.[22] The nanofilled resin used in this study showed smoother surface after brushing with a whitening toothpastes compound with hydrated silica particles as abrasive. Moreover the

association of soft-bristle toothbrush and low abrasive dentifrice did not increase roughness for nanofilled and microfilled materials.[8]

An inadequate oral environment, with episodes of corrosive and abrasive challenges, can promote some properties changes of the composite resin.[22] The hardness of the composite resin is an important property for evaluating the resistance of this material to indentation. However, the interval time between the corrosive and abrasive challenges evaluated, not influence in hardness in this study. The microhardness are related to the composition and content of the particles.[23]

According to all these aspects addressed, the waiting time from acidic beverages intake until performing oral hygiene is important for maintaining the surface roughness of the restoration. Moreover, considering the limitations of this manuscript, future studies should be performed with a longer immersion time, different types of beverages, others composite resins and different toothpaste.

Conclusion

Therefore, with the limitations of this article, it is possible to conclude that the immersion in acidic beverage followed immediately brushing with whitening toothpastes, increased the surface roughness. The 30 minutes between intake of acidic beverages and brushing was important to decrease the deleterious effects of composite resin restauration. Microhardness not influenced by the interval between ingestion of acidic beverage and brushing.

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Figure legends

Figure 1: Experimental designs defined according to time elapsed between corrosive and/or abrasive challenges.

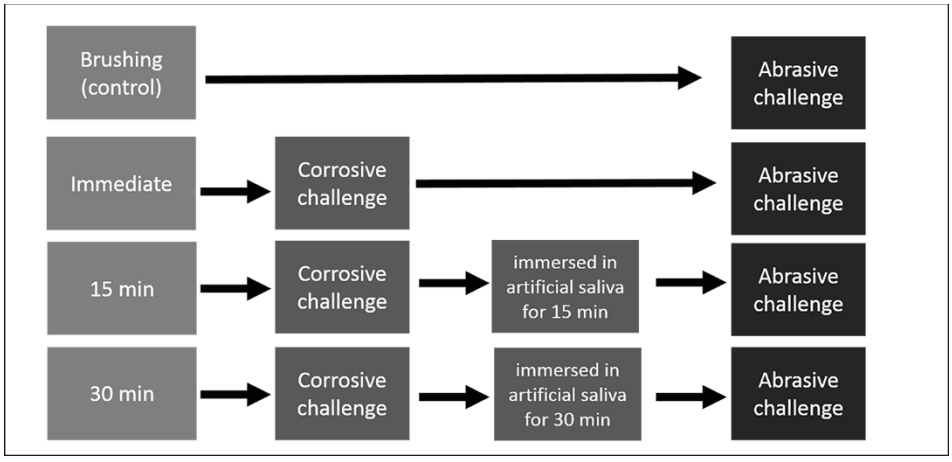
Figure 2: Scanning electronic microscopies (1.000x magnification) illustrating the composite surface before (A) and after corrosive and/or abrasive challenges: (B) only abrasive challenge (control); (C) No-interval between the challenges; (D) interval of 15 min between the challenge; (E) interval of 30 min between the challenges.

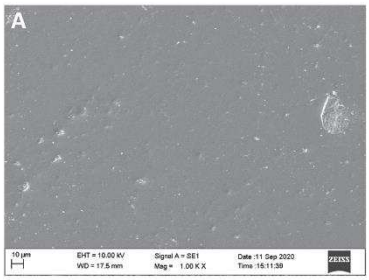
Table

Table 1. Means (standard deviation) of changes on roughness and hardness of composite specimens according to time elapsed between the corrosive and abrasive challenges (n = 9).

Treatments	Roughness (µm x 10^{-2})	Hardness (KHN)
Control*	-9.4 (10.9) ^{A}	-6.1 (14.3)
No-interval	11.7 (14.4) ^{B}	-9.9 (14.3)
15 min.	-3.74 (11.8) ^{AB}	-7.2 (9.2)
30 min.	-5.81 (11.7) ^{A}	-10.3 (13.0)

a-For roughness, distinct letters indicate statistical difference ($p < .05$). For hardness, there was no statistical differences among the treatments. * Only abrasive challenge.

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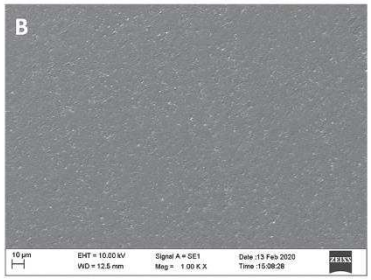
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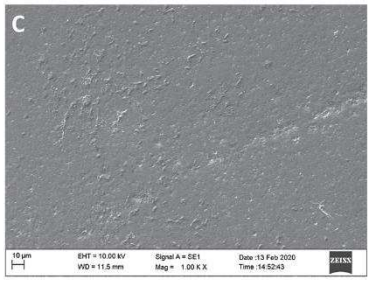
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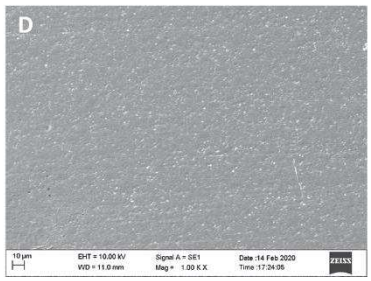
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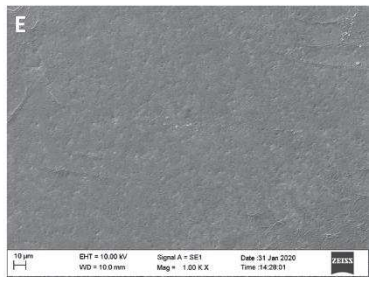
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Capítulo II

Influence of different microshear parameters on bond strength and stress distribution.

Ana Laura Rezende Vilela,[a] Alexandre Coelho Machado,[b] Fernanda Pereira Silva,[c] Grazielly Borges Oliveira, [d] Paulo Vinicius Soares,[e] Luís Henrique Araújo Raposo,[f] Murilo de Sousa Menezes.[g]

a- DDS, MSc, Graduate student, School of Dentistry, Federal University of Uberlandia. Av. Pará, 1720, Bloco 4L, Anexo A, Sala 34, Campus Umuarama, Uberlândia, Minas Gerais, Brazil. Contribution: idea, experimental design, performed the experiments, performed statistical evaluation and wrote the manuscript.

b- DDS, MSc, PhD, Professor, Technical Health School, Federal University of Uberlândia. Av. Amazonas, Bloco 4K, Campus Umuarama, Uberlândia, Minas Gerais, Brazil. Contribution: experimental design, performed a certain test, performed statistical evaluation and proofread the manscript.

c- DDS, MSc, PhD, Professor, Department of Dentistry, School of Dentistry, University of Rio Verde, Fazenda Fontes do Saber, Dentistry School, Rio Verde, Goias, Brazil. Contribution: ideia, hypothesis, experimental design and proofread the manuscript.

d- DDS, Graduate student, School of Dentistry, Federal University of Uberlandia. Av. Para 1720, Bloco 4L, Anexo A, Sala 34, Campus Umuarama, Uberlândia, Minas Gerais, Brazil. Contribution: Performed the experiments in partial fulfillment of requirements for degree.

e- DDS, MSc, PhD, Professor, Department of Restorative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia. Av. Pará, 1720, Bloco 4L, Anexo A, Sala 34, Campus Umuarama, Uberlândia, Minas Gerais, Brazil. Contribution: Idea and experimental design

f- DDS, MSc, PhD, Professor, Department of Occlusion, Fixed Prosthodontics and Dental Materials, School of Dentistry, Federal University of Uberlândia. Av. Pará, 1720, Bloco 4L, Anexo A, Sala 34, Campus Umuarama, Uberlândia, Minas Gerais, Brazil. Contribution: idea, experimental design and proofread the manuscript.

g- DDS, MSc, PhD, Professor, Department of Restorative Dentistry and Dental Materials, Dental School, Federal University of Uberlândia. Av. Para 1720, Bloco 4L, Anexo A, Sala 34, Campus Umuarama, Uberlândia, Minas Gerais, Brazil. Contribution: Coordinator, idea, hypothesis, experimental design and proofread the manuscript.

Correspondence:

Dr. Murilo de Sousa Menezes

Federal University of Uberlândia, School of Dentistry, Department of Restorative Dentistry and Dental Materials, Av. Pará, 1720, Bloco 4L, Anexo A, Sala 34, Campus Umuarama, Uberlândia, Minas Gerais, Brazil 38400-902.

Tel +55-34-3225-8106

e-mail: murilomenezes@ufu.br

Acknowledgments

This work was supported by CAPES/Brazil. The authors are indebted to CPBIO/UFU for equipment support.

Abstract

Purpose: The influence of different microshear parameters such distance between the cylinders and wire loop thickness on bond strength and stress distribution.

Materials and methods: Forty lithium disilicate blocks were etched with 10% hydrofluoric acid, followed by 35% phosphoric acid cleaning and silane coupling. The specimens were randomly divided into four experimental groups (n=10), according to the distance between the cylinders (3 mm – D3.0; and 1.5 mm – D1.5) and the wire loop thickness (0.2 mm – T0.2; and 0.7 mm – T0.7). The microshear bond strength (μSBS) test was performed and failure mode of specimens was determined by stereomicroscopy. The failure mode aspects were analyses with scanning electron microscopy (SEM). The finite element analysis (FEA) was performed according to experimental groups and the results was analyzed by maximum shear stress. The statistical analysis was subjected to Shapiro-Wilk, ANOVA two-way and Tukey test.

Results: The μSBS values was no significantly affected by specimen distance (p=0.865), irrespective of the wire loop thickness; whereas the wire loop thickness presented statistical difference for bond strength (p=0.017). The most common failure mode was mixed for D3.0/T0.2, D1.5/T0.2, D1.5/T0.7 and adhesive for D3.0/T0.7. SEM images showed the groups present difference in the failure mode. FEA showed higher shear stress on the bond interface and on the base of the resin cement for T0.7, regardless of the distance between cylinders.

Conclusion: The μSBS parameters affect the analysis of adhesion, the most appropriate wire loop thickness is 0.2 mm. The distances between the cement cylinders is not relevant for μSBS.

Keywords: Adhesion, Bond strength test, Dental Material, Shear bond strength, Shear bond testing, Finite element analysis.

1. Introduction

The adhesive dentistry include a complex set of physical mechanisms (mechanical and chemical) that allow the union of heterogeneous substrates and materials[2, 5]. The mechanical adhesion consists of the formation of microporosities on the tooth surface or restoration, infiltration of resin monomers, and subsequent polymerization of these monomers in the micropores[2, 5]. The chemical adhesion is based on the interaction between the tooth/restorative material substrate and monomers that have acid functional groups or are chelation promoters[2, 5]. Thus, with the advent of adhesive restorative dentistry and development of products with different compositions and adhesive mechanisms, the determination of the bond strength to dental substrates and dental materials is a topic of great importance and interest for manufacturers, researchers and clinicians[29].

Taking into account the philosophy of minimally invasive restorations and requirement of maximum preservation of tooth structure, there are currently several materials and techniques for achieving adhesion between different dental substrates and materials. Such as, application surface treatments for ceramic adhesion[37], diamond bur roughening or air-borne particle abrasion[40] for composite resin repair[48], universal adhesives[18], self-etching primer[25], self-etching adhesives[34] and self-adhesive restorative materials[24]. For this reason, studies on adhesion still are and will continue necessary, in order to scientifically demonstrate if techniques and materials are adequate for each adhesion procedure. Moreover, *in vitro* studies are important to set up a new product, support and generate more security before *in vivo* studies[32]. Nonetheless, many investigations performed the same methodology for bond strength, however using different parameters, making difficult comparison between results from different studies[32, 38].

Two types of bond strength tests are available - macro and micro. The “macro” approaches comprehend shear and tensile bond strength tests that are performed in specimens with relatively large bond areas (usually 3-6 mm in diameter, approximately 7-28 mm^2). However, bond strength values from studies using “macro” tests has been questioned due to the heterogeneity of the stress distribution at the bonded interfaces, besides the fact that more defects are observed in those specimens[14, 46, 47]. Therefore, “micro” test designs are preferred to minimize the unwanted effects described above. In the “micro” test approaches, bonded cross-sectional areas of $1mm^2$ or less are analyzed, due to a lower probability of having a critical size defects and specimens are easily aligned in a crack opening orientation relative to the applied load[3].

The most common approaches used for bond strength test are microtensile (μTBS) or microshear (μSBS) test desings[3]. However, the μTBS specimens are more susceptible to have failures incorporated at the adhesive interface or at the bonding substrates, due to their dimensions[13, 32]. In addition, the preparation of μTBS specimens is complex, generating failures during the production, due to the sectioning or trimming procedures, which by themselves may induce early microcraking in the specimens[1]. However, the μTBS approach is better to determination of bond strengths to erosion/abrasion cavities in tooth substrates or box-like cavities[1].

The μSBS is an extremely useful test for testing bond strength to substrates or materials with more heterogenic properties such as ceramics, composite resins and resin cements [19]. This test design (μSBS) make these materials particularly less susceptible to specimen preparation effects and testing conditions observed in the μTBS approach[20]. The μSBS test design generally involves the application of a lateral loading force on cylinder specimens[28]. With the μSBS test, it is possible to obtain multiple specimens per substrate, however there is no consensus about the

minimum secure distance between the cylinder specimens to avoid unwanted stress to reach the other passive cylinders while loading the active specimen.

In the μSBS test, the loading forces should be applied as close as possible to the bond interface area to promote severe stress concentrations on the desired test site[13]. Initially, loading to promote shear stress was applied with knife edge or point devices[17, 22]. However, these loading devices were replaced, since using a wire loop rather than a knife edge for shear bond tests was shown to reduce the stress concentration magnitude adjacent to the interface and increase microshear bond strength[14, 38]. However, a variety of orthodontic wires thickness is available in the market and this makes more difficult to standardize the micro-shear test. The literature is also scarce in relation to the influence of wire loop thickness on the bond strength results obtained by the microshear test.

Therefore, the aim of this study was to evaluate the influence of different μSBS test parameters such as distance between specimens and wire loop thickness on bond strength and stress distribution. The null hypothesis was that the different parameters tested would not influence the microshear bond strength results and stress pattern distribution.

2. Materials and Methods.

2.1. Specimen preparation

Forty glass-reinforced lithium disilicate ceramic blocks (IPS e.max CAD, Ivoclar Vivadent, Schaan, Liechtenstein) were used as the μSBS substrate. The metal connector was removed from the blocks and then, the ceramic blocks were crystallized by a specific ceramic furnace (Programat® EP3010, Ivoclar Vivadent) for 20 min at 840°C (1544-1562°F). The crystallized ceramic blocks were included in PVC cylinders with polystyrene resin (Aerojet, Santo Amaro, SP, Brazil). After the inclusion, the ceramic surfaces were polished with silicon carbide papers (#600, #800, #1200, Norton, Campinas, SP, Brazil) for 20 s each, to standardize the specimens.

The surface treatment of the ceramics was performed with 10% hydrofluoric acid etching (Condac Porcelana, FGM, Joinville, PR, Brazil) for 20 s, followed by air-water spray washing for 1 min and air-drying for 30 seconds. Subsequently, 37% of phosphoric acid (Condac 37, FGM) was used for cleaning during 1 min, washed with air–water spray for 1 min and air-drying for 30 seconds. Finally, silane coupling agent (Prosil, FGM) was actively applied on the ceramic surface, waiting 60 s for react, followed by light air-jet for 5 seconds[41].

After, the ceramic specimens (n=10) were randomly divided into four experimental groups as described in Table 1. Resin cement cylinders (RelyX U200, 3M ESPE, St Louis, MO, USA), were made with tygons tubing (0.8 mm Ø and 1 mm height; TYG-030, Small Parts Inc., Miami Lakes, FL). Six specimens were obtained per substrate with distances between then of 1.5 mm and 3 mm, according to the experimental groups. The autoadhesive dual-cure resin cement was manipulated according to the manufacturer's instructions and after 5 min of mixing, the cement was light-cured for 40s on each face with a LED device (Radii Plus, SDI, Victoria, Australia) with average irradiant intensity of 1400 mW/cm². Finally, the tygon tubing was removed with a scalpel blade and the specimens were stored in 100% humidity at 37°C for 24 hours.

2.2. Microshear bond strength test (μSBS)

The specimens were attached to a specific device fixed to a universal testing machine (EMIC DL 2000, São José dos Pinhais, PR, Brazil) so the long axis of the cylinders was perpendicular and the adhesive interface parallel to the vertical plane. According to the experimental groups, 0.2 mm or 0.7 mm- diameter wire loop (NiCr, Morelli, Sorocaba, SP, Brazil) was placed around the base of the resin cement cylinder over the ceramic/resin cement adhesive interface. A 20kgF load cell was used to apply an increasing parallel force to the adhesive area (material interface) at a speed of 0.5 mm/min, until specimen failure occurred. The bond strength (MPa) for each

resin cylinder specimen was calculated according to the following formula: $\tau = F/ \pi r^2$, where F is the force required for failure (N) and πr^2 is the bonded area (mm^2) of the specimens[11].

2.3. Failure mode analysis

The failure mode of the specimens was analyzed by stereomicroscopy (Mitutoyo Kawasaki, Japan) at 40× magnification. Failure modes were categorized as the following types: I- adhesive interfacial failure between ceramic and resin cement; II- cohesive failure in ceramic; III- cohesive failure of resin cement; IV- and mixed (adhesive-cohesive) failure[11].

2.4. Scanning electron microscopy (SEM)

After failure mode analysis, representative specimens of each group were submitted to SEM analysis (EVO MA 10, Carl Zeiss, London, UK) at a voltage of 20 kV. After desiccation, the specimens were fixed on metal stubs and them were sputtered with gold (1 cycle of 120 s) under vacuum in a sputtering device. The surface was analyzed by SEM at a magnification of 80X after the microshear test in order to better illustrate the failure sites[36].

2.5. Finite element analysis

Tridimensional (3D) finite element linear elastic analysis was performed using geometric representations for the microshear test design. Four computer aided design (CAD) models were generated using CAD software (Rhinoceros 4.0 3D software, Rhinoceros, Miami, FL, USA), simulating the experimental groups and the different conditions evaluated in the microshear test (Fig. 1).

The CAD models were exported to the Finite element analysis (FEA) software (ANSYS 18.2, Ansys Workbench 18.02, Canonsburg, PA, USA) using the Standard for the Exchange of Product DATA (STEP) format. The mechanical properties of each structure were defined as homogenous and isotropic: lithium disilicate (E = 95 GPa[12, 26] v = 0.3[8]); resin cement (E = 18.6 GPa[45], v = 0.35[45])

and nickel–chrome (E = 188 GPa[17], v = 0.28[17]). After testing the mesh conversion to define the appropriate mesh refinement level, volumes corresponding to each structure were meshed with the controlled and connected solid quadratic tetrahedral elements of 10 nodes (Fig. 2A). For standardization, the load application of the finite element models was obtained by averaging all the maximum loading data obtained by the four experimental groups in the microshear test (29.985 N). The load was applied on all surfaces of the wire loop, parallel to the interface and perpendicular to the long axis of the resin cement specimen (Fig. 2B). The models were constrained at the base and lateral surfaces of lithium dissilicate blocks (Fig. 2C). The stress distribution analysis was performed using maximum shear stress criterion and plotted according to the CAD geometry. The specific shear stress values from the cylinder tested (active) to the end of the adjacent cylinder (passive), were also measured and plotted on graphs[27].

2.6. Statistical analysis

Data for microshear bond strength was first subjected to the Shapiro-Wilk test for testing normality. Subsequently the values were submitted to two-way analysis of variance (ANOVA) and Tukey's test. The level of significance was set in 5% and all the analyses were performed using a statistical software package (SigmaPlot version 12.0, Systat Software Inc., San Jose, CA, USA).

3. Results

3.1. Microshear bond strength and failure mode

The mean microshear bond strength (μSBS) values (MPa) for the experimental groups are shown in Table 2. According to the two-way ANOVA, the factor specimen distance showed no significant effect (p=0.865), irrespective of the wire loop thickness; whereas the factor wire loop thickness presented significant effect (p=0.017) for both specimen distances evaluated. No significant interaction was found between these factors (p=0.903). The microshear bond

strength values found for the 0.2 mm wire loop thickness were significantly higher than those of 0.7 mm.

The failure modes verified for the experimental groups are shown in Table 3. Mixed failure mode was the most commonly found (Type IV), except for group D3.0/T0.7, which present prevalence of adhesive failures (Type I). The adhesive failure mode was the second most common, among all groups. No cohesive failure in ceramic was found (Type II). The adhesive failure in resin cement (Type III) present intermediary percentage of failure mode.

3.2. Finite element analysis

The maximum shear stress distribution verified for all simulated μSBS conditions is presented in Figures 4 to 7. No differences were observed in the shear stress pattern at the adhesive interface of the adjacent resin cement cylinder specimen (passive), independently of the wire diameter or the specimen distance evaluated (Figs. 4 and 7). The shear stress distribution observed from the adhesive interface of the tested specimen (active) to the adjacent resin cement cylinders (passive) was similar for the D1.5 and D3.0 models (Fig. 7). Irrespective of wire loop thickness, the maximum shear stress verified in the passive specimen region was lower than 1.42 MPa for the D1.5 models and 0.34 MPa for the D3.0 models.

The variation in wire loop thickness resulted in different shear stress distribution on the adhesive interfaces between the tested resin cement cylinder (active) and ceramic block, regardless of the specimen distance evaluated (Figs. 4, 5 and 6). For the 0.2 mm wire loop diameter, the shear stress was found concentrated mainly on the interface area where the wire made direct contact to the tested resin cement cylinder specimen. For the 0.7 mm wire loop diameter, the stress was concentrated on all the peripheral margin of the ceramic and resin cement adhesive interface (Fig. 5). Moreover, the thicker wire showed greater shear stress values on the interface area compared to the thinner wire. Irrespective of the specimen distance

evaluated, the shear stress on the interface of the tested resin cement cylinder specimen (active) exceeded 70 MPa for the T0.7 models and 40 MPa for the T0.2 models. On a longitudinal section perspective, it was possible to note higher stress concentration on the cylinders of the T0.7 models, from the interface until its contact with the wire (Fig. 6).

3.3. Scanning electron microscopy

SEM analysis illustrated the failure modes observed in the specimens after the microshear bond strength test. The SEM images showed that specimens tested with the 0.2 wire loop, presented less cement residues on the center and margin of substrate than specimens tested with 0.7 wire loop. Moreover, the SEM images of the failure sites of specimens were compatible with the FEA findings at the interface (Fig. 3).

4. Discussion

Although the distinct distance between specimens have not influenced experimental and computational results, the different wire loop thickness affected microshear bond strength values and stress distribution pattern. Therefore, the null hypothesis tested in the present study was rejected, since the μSBS test parameters evaluated were influenced.

The microshear bond strength is one of the most commonly employed test designs for evaluating bond strength of dental materials to different substrates[23], for being a test of rapid development, which is inexpensive for testing routines in most dental school or research laboratories[35]. Beyond the advantages of the microsehar bond test to compare performance of new and/or experimental material and technique[16, 45, 46], this approach remains a useful method for testing brittle substrates, as enamel and ceramics, that are particularly susceptible to preparation artefacts such as those related to the microtensile bond strength method[33]. Despite these advantages, the microshear test presents a great limitation: absence of standardization

during the specimens preparation and the load device application, which make difficult to discuss and compare the results of previous and new researches on bond strength[35].

Many investigations confectioned several cylinders in the same specimens[9, 15, 20], without worrying about the distance between them and if this distance would generate residual stress in the adjacent cylinders (passive). However, in the present study, the distance evaluated between specimens of 1.5 or 3.0 mm showed no statistical difference on bond strength and stress pattern was similar also in FE results. Nonetheless, specimens with less than 0.5 mm distance between then, can generate undesired stress to reach adjacent specimens. This fact may impact the bonding strength of these cylinders to the substrate, since stresses are reaching their adhesive interface even before they are tested, what can lead to unreliable bond strength values. Moreover, a previous investigation showed a high stress mainly close to the region of load application[14], what was also confirmed by the present study.

The gripping devices are the site where specimens are attached to mechanical testing machines. Specimens must be positioned parallel and aligned with the loading application devices of testing machines. When the specimen is not positioned parallel to loading application direction, the bonding interface will be not perpendicular to the surface and the applied force may be uneven distributed to the specimen. Due to the non-ideal loading conditions, either the specimen or gripping mechanism can significantly alter the stress distribution at the bonded interface[39]. The wire loop is a type of loading application device, and the 0.2 mm thickness presented better results than the 0.7 mm as showed by the μSBS values and FE analysis. The distance between the interface and contact point between the wire and the resin cylinder depend on the wire diameter, and as seen, it can affect the stress distribution at the bonding site. The 0.7 mm wire loop increased the distance between the wire contact point and resin

cylinder (bonding interface). This situation occurs because of the increase of the flexion moment value by distancing the load application point in relation to the bonding interface[38].

Considering the current literature, available normative and published theses, there is no standardization on the thickness of orthodontic wires used for μSBS tests. According to some investigations, the wire loop thickness more commonly used was 0.2mm or 0.3mm[7, 10, 20, 43]; however, some studies used different wire thickness[38] or have not described which wire loop thickness and gripping device type used[6, 19, 30, 42].

In relation to the failure mode, the mixed failure were predominantly found in almost experimental groups, except D3.0/T0.7. These results can be explained by FE analysis (Fig. 5), in which different stress distribution was verified in the ceramic substrate where the cylinders were placed. The stress distribution among groups present similarity with SEM failure mode. Other studies have also shown predominance of mixed failure mode when performing microshear bond strength test[19, 20, 43, 44].

Parameters for specimen preparation and performing μSBS methodology have already been standardized in the literature[31]. Specimens were prepared in a standardized approach using tygon tubing for this purpose, which is a method frequently used for the preparation of specimens for microshear tests[4, 25, 28]. One of the main benefits of this approach is that great control over the dimensions of the adhesive interface is possible[16]. However, if not performed correctly, it can generate bubbles, flaws or disturbs in the cylinders when removing the tygon matrixes, making specimens inadequate to test[17]. Finally, the crosshead speed used for the test was set in 0.5 mm/min, since previous studies showed that a crosshead speed of 0.5 mm / min or 0.75 mm / min present no statistical differences when used in shear and tensile test designs[21]. Higher test speeds can generate outside loading of the adhesive interface, with load application

on the restorative materials and/or substrates, thereby increasing cohesive failures in place of adhesive/mixed failures[21].

Despite considerable advances regarding bond strength tests, limitations still exist on the current methods. Besides that, the lack of standardization requires new bond strength testing procedures. The standardization of the parameters of bond strength tests is of great importance for a better comparison of manuscript's data and greater dissemination of the knowledge about the adhesive materials and the adhesion methods[33,31]. Therefore, the standardization of μSBS wire loop with 0.2 mm diameter may be important step for better distribution of stress in the adhesive interface as shown. Finally, superior distances to 1.5 mm between the cylinder specimens is safe to avoid undesirable stress to reach adjacent specimens in μSBS test design.

5. Conclusion

Within the limitations of the present study, it may be concluded that the diameter of wire loops influenced the microshear bond strength results and the distance between the cylinder specimens seems not relevant when superior to 1.5 mm. In order to standardize, it is recommended to use 0.2 mm diameter wire loop and 1.5 mm or superior distances between the cylinder specimens.

Clinical Relevance

More standardized laboratory bond strength tests allow more reliable results for clinical inference.

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Table

Table 1

Characteristics of the experimental groups according to distance between cement cylinders (specimen distance) and wire loop thickness (n=10).

	Distance between specimens	**Wire loop thickness**
D1.5/T0.2	1.5 mm	0.2 mm
D3.0/T0.2	3.0 mm	0.2 mm
D1.5/T0.7	1.5 mm	0.7 mm
D3.0/T0.7	3.0 mm	0.7 mm

Table 2:

Table 2:

Mean microshear bond strength (μSBS) values (MPa) and standard deviation (±) according to the distance between specimens (D) and wire loop thickness (T).

	T0.2	**T0.7**
D1.5	20.6 ± 5.1 Aa	16.8 ± 4.3 Ba
D3.0	21.0 ± 5.8 Aa	16.9 ± 4.0 Ba

Lowercase letters indicate significant differences between columns (vertical): specimen distance (D) (p=0.865). Uppercase letters indicate significant differences between rows (horizontal): wire loop thickness (T) (p= 0.017).

Table 3:

Failure mode distribution (%) among the experimental groups.

	D1.5/T0.2	D3.0/T0.2	D1.5/T0.7	D3.0/T0.7
Type I - Adhesive failure	26.6%	41.6%	33.3%	56.6%
Type II - Cohesive lack in ceramic	0%	0%	0%	0%
Type III - Cohesive failure in resin cement.	6.6%	11.6%	10%	10%
Type IV - Mixed failure	66.6%	46.6%	56.6%	33.3%

Figures

Figure 1:

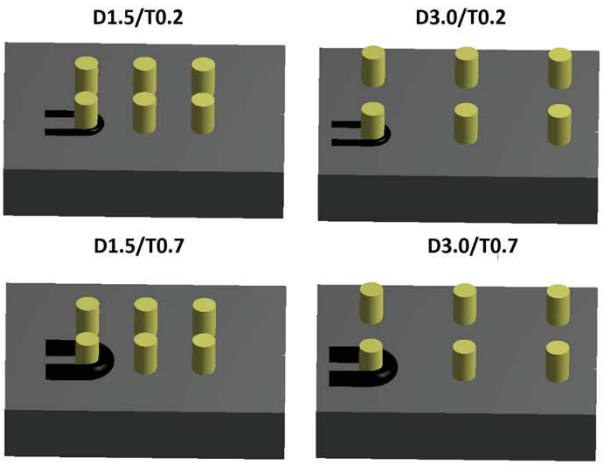


Figure 2

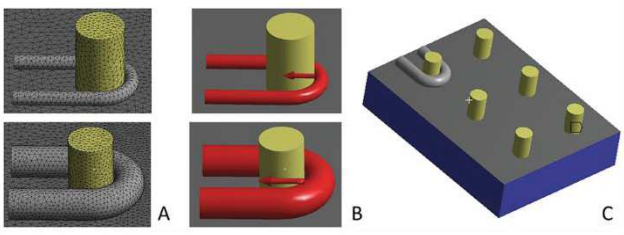


Figure 3:

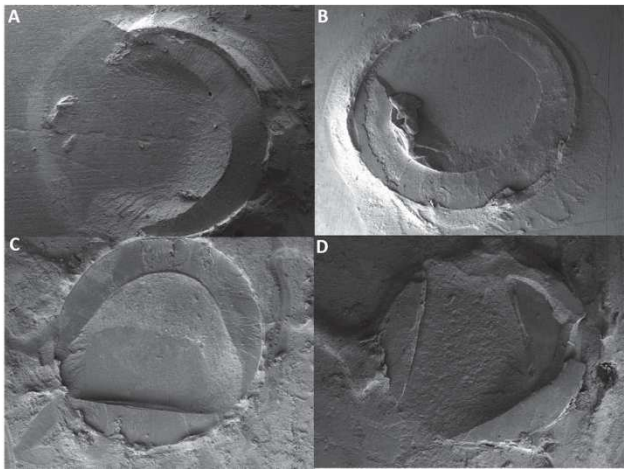


Figure 4:

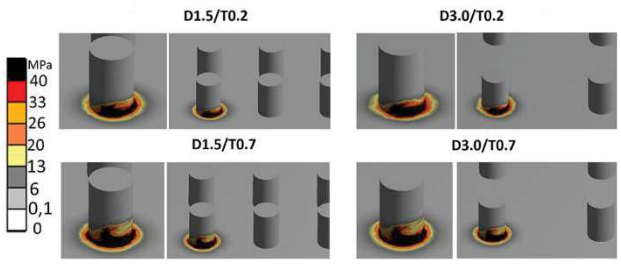


Figure 5

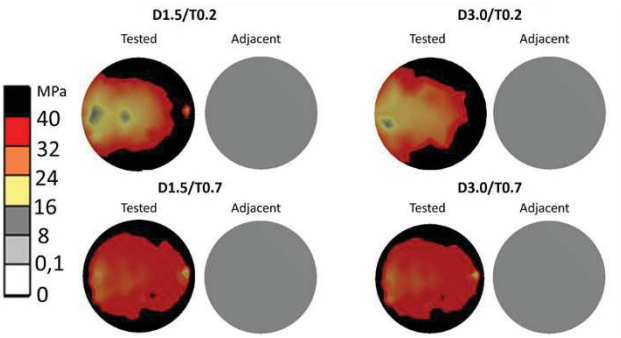


Figure 6

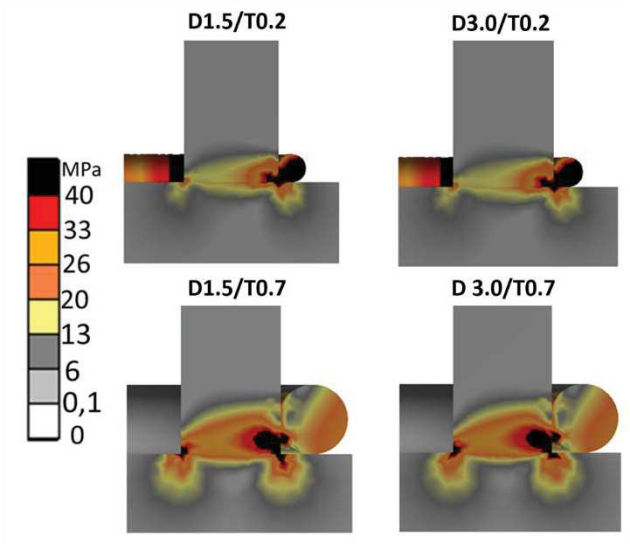
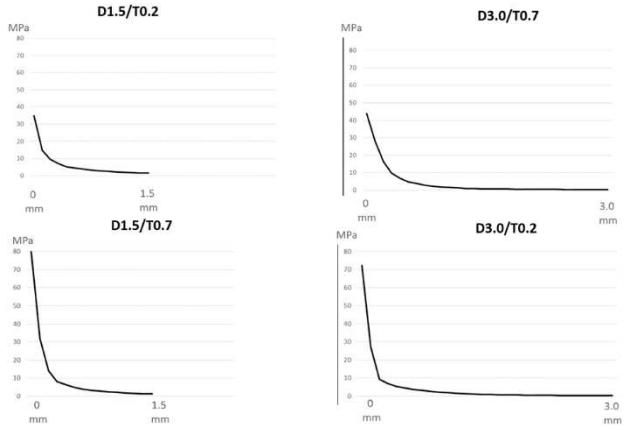

D1.5/T0.2
D3.0/T0.2
MPa
40
33
26
20
13
6
0,1
0
D1.5/T0.7
D 3.0/T0.7

Figure 7


D1.5/T0.2
MPa
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D3.0/T0.2
MPa
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Legends:

Figure 1: CAD models simulating the different μSBS test conditions, according to distance between the specimens and the wire loop thickness.

Figure 2: Pre-processing of the the finite element analysis. A: Meshing of the models. B: Loading applied to simulate the microshear test. C: Boundary conditions: the displacement of the model is null on the blue area.

Figure 3: Scanning electron microscopy images of the failure sites after performing the μSBS test for the experimental groups: (A) D1.5/T0.2; (B) D3.0/T0.2; (C) D1.5/T0.7; (D) D3.0/T0.7).

Figure 4: Stress distribution by maximum shear stress criterion (MPa) on tested (active) and adjacent (passive) cylinder specimens for all the experimental conditions. The colors closer to black represent the highest shear stress values, while the colors closer to white represent the lowest shear stress.

Figure 5: Stress distribution by maximum shear stress criterion (MPa) on the bonding interface of the tested (active) and adjacent (passive) resin cement cylinder specimens and ceramic for all the experimental conditions. The colors closer to black represents the highest shear stress values, while the colors closer to white represents the lowest shear stress.

Figure 6: Longitudinal section perspective of stress distribution by maximum shear stress criterion (MPa) on the tested resin cement cylinder (active) specimen, ceramic and wire loop for all the experimental conditions. The colors closer to black represents the highest shear stress values, while the colors closer to white represents the lowest shear stress.

Figure 7: Shear stress values (MPa) on the ceramic surface according to the distance between the tested (active) and adjacent (passive) cylinder specimens.

Capítulo III

Influence of silane open time on bond strength and surface energy in resin composite repair.

Effect of the opened silane in composite repair

Ana Laura Rezende Vilela[a], Marcela Gonçalves Borges[a], Letícia Raquel de Oliveira[b], Washington Martins da Silva Junior[b], Alexandre Coelho Machado[c] Murilo de Sousa Menezes[a]

a- Department of Operative Dentistry and Dental Materials, Dental School, UFU - Universidade Federal de Uberlândia, Uberlândia, MG, Brazil.

b- School of Mechanical Engineering, UFU- Universidade Federal de Uberlândia, MG, Brazil

c- Health Technical School, UFU - Universidade Federal de Uberlândia, Uberlândia, MG, Brazil.

Corresponding author:

Prof. Dr. Murilo, de Sousa Menezes

Federal University of Uberlandia, School of Dentistry, Department of Restorative Dentistry and Dental Materials. Avenida Pará, 1720, Bloco 4L, Anexo A, Sala 34, Campus Umuarama Uberlândia - Minas Gerais – Brazil, Tel.: +55 34 3225-8105; Fax.: +55 34 3218 2279 E-mail address: murilomenezes@ufu.br

Influence of silane open time on bond strength and surface energy in resin composite repair.

Effect of the opened silane in composite repair

SUMMARY

This study evaluated the efficiency of open silanes vials stored at room temperature (22ºC) or at 5ºC on the bond strength between composite resins. Ninety nanohybrid composite resin disc-shaped were prepared and included in a polystyrene resin. The specimens were aging for 4 months in distilled water at 37 ºC. The silanes, pre-hydrolised (PH) and immediate hydrolysis (IH) was stored at room temperature (RT) or low temperature (LT) for a year. The silane's vial was opened three times a week during 1 year to simulate the clinical use, and the new silane used as received (AR) was not aged. The composite resins specimens were blasting with aluminum oxide for the surface treatment asperization, cleaned with phosphoric acid and then the silane and adhesive system were applied. four specimens were performed per disc-shaped. The specimens were submitted to the microshear bond strength (μSBS) and contact angle (CA) tests. The failure mode was analyzed on the Scanning electron microscopy-(SEM). The statistical analysis was performed by Two-way ANOVA, Tukey's tests and Kruskal Wallis (α = 0.05). The μSBS values were higher for the PH-NS (14.54), however decreased the values after aged, regardless of storage type. The IH-NS (12.14) showed lower values of μSBS than PH-NS, however maintained the adhesion values after aging at room temperature (IH-RT: 13.43). The CA test showed no difference in storage (P = 0.054) and the PH showed lower CA than IH group (P = <0.001). The failure mode showed more adhesive and cohesive failure in composite resin base. Therefore, it was concluded the silane aged influence in bond strength. The room temperature maintains the characteristic of silane with immediate hydrolysis.

Key Words: silanes, adhesion, dental materials, bond strength, composite resins.

INTRODUCTION

Productive time and conservative restorations are the wishes of the patient and the dentist. For this reason, resin composite restorations are the material of choice for most rehabilitation. Resin composite restorations show excellent results when performed freehand, that is, without the need for laboratory steps, which also generates lower cost of the procedure for the patient and the dentist (1). The aesthetics of this restorative material is similar to natural teeth, due to the various enamel, dentin and effect resins (2). In addition, the resin composite presents a elasticity modulus similar to that of dentin, which gives a homogenous distribution of the stress generated by chewing (3).

The annual failure rate of resin composite restorations varies from 1 to 4% in permanent teeth (4, 5). The main failures of restorations are fracture, secondary caries and changes in the restorations esthetics (staining and roughness) (5). Most of these failures can be repaired instead of replacing the restoration, preserving the tooth structure, reduction of treatment costs and delaying the "restoration death spiral". The resin composite repair presents a satisfactory longevity than restorations that have been completely replaced (6, 7). However, the remaining parts of the original restoration showed a higher success rate than the repaired part (7) and the lower success rate of repairs might be related to bonding failures (7).

The adhesion of composite repairs occurs in two ways: micromechanics and chemical adhesion. Micromechanical adhesion consists of new composite adhesion to aged composite through micromechanical interlocking to irregularities in the surface roughness. This micromechanical adhesion in aged resin composite restorations is created by asperization with diamond burs or by sandblasting with aluminum oxide. The sandblasting with aluminum oxide is the most performed, due to the promising bond strength results in the literature (8). Chemical adhesion in resin composite repair is created by the silanes bonding agents with filler particles and organic matrix (9).

Silane coupling agents are also widely used for glass ceramic bonding, fiber glass post bonding and repair of restorative resin composite (10). The application of silane is an important step and cannot be disregard. Silane is a bifunctional molecule that may also

form covalent bond with filler particles and co-polymerize with methacrylate groups present in repair resin composite. MPTMS (ϒ-methacryloxypropyl trimethoxysilane) is the most commonly silane used in dental applications, since its methacrylate functionality matches that of most dental resins (10). The silanol group present in the silane coupling agent reacts with glass surface forming a siloxane bonding. In addition, silanes increase surface wetting, thereby enhancing diffusion of the bonding agent into the substrate. The silane pre-hydrolysed (pre-activated) was composed with silane monomer diluted and dissolved in ethanol, water and acetic acid for pH adjustment. The silane concentration varies between ca. 1–10 vol% in different commercial products. However, when the pre-activated silane's bottle is opened for the first time, as it is very unstable, has a reduced useful life and loses its adhesion efficiency (10). This is due to the excess formation of siloxane oligomers/polymers that are inactive (10). On the other hand, immediate hydrolysis silanes present a prolong the shelf life. This system present two vials. The one vials contain unhydrolysed silane monomer dissolved in ethanol and the other one contains aqueous acetic acid. The last mixed immediately before use to allow hydrolyze silane (10).

Therefore, this study aimed to analyze the efficiency of different silanes (pre-hydrolyzed and immediately hydrolyzed stored at room temperature (22ºC) or at 5ºC in bond strength between resin composite. The null hypothesis was that the silane type and storage methods would have no effect on bond strength in resin composite repair.

METHODOLOY

Two Silanes coupling agent, Pre-hydrolysed (Prosil-FGM, Joinville- SC, Brazil) and Immediate hydrolysis (Silane- Dentsply Ind e Com. Ltda., Petropólis RJ, Brazil), were evaluated in three ways (as receved (no storage), 1 years after storage in refrigerator and 1 years after storage in room temperature). The silanes were evaluated by microshear bond strength, contact angle and failure mode.

Silane storage

The silanes were aged in two ways: in a cabinet at room temperature (22ºC) and in a refrigerator (5ºC) for 1 year. To simulate the clinical use of silane, the bottles were opened

3 times, in different days by a week, for 90 seconds. The new silane (AR)were used for simulate immediate use after opening the vials.

Resin Composite specimens

A total of ninety disc-shaped specimens (n=12) of nanohybrid resin composite (Herculite Preciss, Kerr, Orange, CA, USA), shade A3 were prepared (6mm in diameter and 1mm in thickness) in a teflon cylindrical matrix under a glass plate. The cylindrical mold was covered with mylar strip. After that, a pressure was applied, for 10 seconds, to extrude excess of resin composite and to obtain a smooth and flat surface in each specimen. The specimens were irradiated on both sides for 20 s using light-aticvated polymerization unit (Valo, Ultradent, Indaiatuba, SP, Brazil – 1.000 mW/cm², according the manufacture). After polymerization, the mylar strip and the glass plate on the top of the mold were removed. The specimens were stored in distilled water at 37ºC. After 24 hours, specimens were embedded in polystyrene resin (Aerojet, Santo Amaro, Brazil) and finished with sequential silicon carbide sandpapers, in increasing order of granulation (#600, 800, 1200 and 2000) under irrigation (Politriz Universal, Arotec, São Paulo, SP, Brazil). Then, the specimens were cleaned in an ultrasonic with distilled water for 10 min and stored in distilled water at 37 ºC for 4 months. The distilled water of the specimens was changed weekly.

Specimens preparation

After 4 months, the specimens were randomly divided into six groups according to the type of silane and storage. The materials used and their chemical compositions are listed in Table 1. The sandblasting was conducted with a sandblaster device (Bio-Art, São Carlos, SP, Brazil) with 50 μm aluminum oxide particles (Al_2O_3). The nozzle was hold perpendicular to the surface for 10s at a distance of 10mm. 37% phosphoric acid (Condac 37, FGM, Joinville, SC, Brazil) etchant was applied for 30s and after were rinsing for 30s for cleaning purposes. Humidity was removed with air jets for 30s. Silane coupling agent was applied according to the manufacturer and actively applied using microbrush applicator and volatilization was expected for 60s. A layer of bond agent (Âmbar APS, FGM, Joinville- SC, Brazil) was applied, air-thinned and light-cured for 20s.

Micro-shear bond strength (μSBS) test

For simulate the resin composite repair restorations, tygon tubes (TYG-030, Small Parts Inc., Miami Lakes, FL, USA) were used with an internal diameter and height of approximately 1.70 and 1.50mm, respectively. The Tygon tubes (4 tubes for each specimen) were placed with a minimum distance of 1.5mm between then and over the aged resin composite. The repair resin composite was the same of aged resin composite specimens. The cylinders were photoactivated for 20s, and then the molds were cut longitudinally with a scalpel blade and carefully removed by the same operator. The specimens were stored at 37ºC for 24h in distilled water before the test.

A caliper (Mittutoyo 530312B10, Tokyo, Japan) was used to measure the dimensions of each resin cylinder used to simulate the repairs before the mechanical test. An orthodontic wire with a diameter of 0.2mm (Morelli, Sorocaba, SP, Brazil) was placed perpendicular to the load axis of the resin tubes, which were parallel to the horizontal plane. A 50N load cell was used to apply an increasing parallel force to the adhesive area. Bond strength was tested using a mechanical machine (Microtensile Machine OM 100, Odeme) with a crosshead speed of 1 mm/min until specimen fracture occurred. The bond strength (MPa) of each specimen was calculated according to the following formula: $T=F/A$, where kgf is the force required for failure (N) provided by the machine and πr^2 is the bonded area (mm^2) of the specimens.

Failure mode analysis

The failure area was examined by stereomicroscopy (Mitutoyo, Kawasaki, Japan) at 40x magnification, to assess the failure modes. There was no premature failure. Failure was classified by two evaluators as adhesive failure, cohesive failure in aged resin composite, cohesive failure in repair resin composite or mixed failures. The adhesive failures occur at the adhesive interface and cohesive when partial fracture occurs on composite resins. The mixed failure occurs in interface and one of the resin composite.

Scanning electron microscopy (SEM)

Representative specimens (n=2) of each group were selected to evaluate the effects failure mode. The specimens were analyzed under scanning electron microscope (SEM Leica EM SCD50, Leica Microsystems, Wetzlar, Lahn-Dill, Germany) at 15.0 kV, after sputter-

coating with a thin film of gold. The SEM photomicrographs were taken with 25x magnification for qualitative analysis of the specimens.

Contact angle test:

CA were measured to investigate the wettability characteristics of the silane coupling agent. The silane was subjected to CA analysis with a goniometer using the sessile drop technique. The distilled water was used in the room temperature. The wettability was quantified through the application of a distilled water micro drop through the Goniometer (Ramé-Hart, Inc. Modelo NRL A-100). To carry out the methodology, equal volumes (0.5µl) of liquid were released on the treated surface of the resin composite at three different points. The images were captured using a high-focus digital camera and analyzed by the ImageJ software (Wayne Rasband, National Institutes of Health, Rockville, MD). The angles were photographed 20s after the drop came into contact with the sample. Quantifications of wettability were made in 03 samples according to the experimental groups, totaling 18 measurements. The mean and standard deviation of the CA values found were calculated.

Statistical analysis

The µSBS and CA were tested for a normal distribution (Shapiro-Wilk) and for equality of variances (Levene test), followed by parametric statistical test. Two-way analysis of variance (ANOVA) was performed for µSBS and CA. Multiple comparisons were made using the Tukey test. The frequency distribution of failure pattern was compared with Kruskal-Wallis test. All the tests employed $\alpha = 0.05$ significance level, and all the analyses were carried out with the statistical package Sigma Plot version 13.1 (Systat Software Inc, San Jose, CA, USA).

RESULTS

Micro-shear bond strength

The mean values and standard deviations of µSBS are described in Table 2, showing statistically significant difference between experimental groups. The statistical

analysis showed the interactions between “silane type” and “storage type” (P = <0.001). The “PH” showed highest bond strength for “AR” than “RT” (p<0.001) and “LT” (p<0.001) and “LT” and “RT” there was no statistical difference (p=0.990). The “IH” showed highest bond strength for “AR” than “LT” (p<0.001) and “RT” than “LT” (p<0.001). The “AR” and “RT” there was no statistical difference (p=0.182). For “AR” the “PH” showed higher bond strength than “IH” (p=0.005). For “RT” the “IH” showed higher bond strength than “PH” (<0.001). For “LT” there was no statistical difference for “PH” and “IH” (p= 0,010).

Failure Mode:

The failure mode is described in Table 3. The most prevalent type of failure was adhesive and cohesive in resin composite base (aging composite). Comparing the failure distribution, Kruskal Wallis analysis (Table 3) showed that statistical difference for IH-RT and PH-AR than other experimental groups.

Scanning electron microscopy

The qualitative specimens of each group after the failure pattern are represented by figure 1. As reported in table 3, the majority of failures in the specimens were cohesive in resin composite base, adhesives or in a smaller number mixed failures (involving base resin cohesive and adhesive).

Contact Angle:

The mean values and standard deviations of CA are described in Table 4. The statistical analysis showed the difference among the Silane Types (P=<0.001) “PH” present lower CA than “IH”. However, the “storage type” not presented statistically difference among groups (p= 0,054).

DISCUSSION

Pre-hydrolyzed silanes (single bottle) show higher bond strength values when used immediately after opening compared to immediate hydrolysis silane. However, pre-hydrolyzed silane become unstable after one year regardless of the stored location. The immediate hydrolysis silanes (2 bottles) have lower bond strength; however, if stored in

a room temperature, they are more stable over time. Therefore, the null hypothesis that the silane type and storage method would have no effect on bond strength in resin composite repair were reject.

Aging resin composite restorations may have several characteristics that require restoration repair or replacement, such as fracture, staining and shape changes (5). For repairs procedure, it is necessary to perform a physical and chemical surface treatment for adhesion between the new resin composite and old resin composite (11). Because, the adhesion between resin composite is carried out by free radicals present on the surface of the restoration (12). However, the old resin composite no longer has the free radicals, due to sorption, solubility and leaching actions of the surface layer on the resin composite (13). The silane primer is one of the materials used in the surface treatment of restorations responsible for chemical adhesion (10, 14, 15).

Silane must be activated before application on the restorative material (10). The activation is performed with hydrolysis in an aqueous media, which may be water, alcohol or acetone with a pH of approximately 4 (10). Silane is hydrolyzed to form silanois (Si-OH). These, will react with the silica present in restorative materials forming the siloxane (Si-O-Si) bonds through a condensation reaction (10). There are two forms of hydrolysis of silane primes: pre-hydrolysates (one bottle) and immediate hydrolysis (2 bottles). Pre-hydrolysates have a short half-life after opening (10). This occurs because the pre-hydrolyzed silanes have a high rate of hydrolysis (16, 17). However, the down side is that the formation of oligomers could reduce the effectiveness of the solution in the longer term (17, 18). This result can be confirmed by the bond strength values of the pre-hydrolyzed silanes, in which they presented higher values of bond strength soon after opening, but reduced the resistance by 36% after an open year. The oligomers created by the self-condensation silanol must have resulted in a significant decrease in bond strength in previous studies (17, 19). These results demonstrated the pre-hydrolyzed silane may have lost its initial activity during a long storage time because of the auto polymerized silane oligomers (19). When negative dehydration self-condensation occurred among the silanols of neighboring molecules, they formed oligomers, potentially reduced the long-lasting effectiveness of silane solution (19).

Immediate hydrolysis silane have to be mixed in order to initiate the hydrolysis reaction and the results of this study showed that they are more stable after one year of use when stored at room temperature. The optimum time for silanization was after hydrolysis and before oligomerization because silane highly effective at this period and it can be easily adsorbed as a monomer (19). In another hand, the imediate silano present a lower bond strength soon after opening than pre-hydrolyzed silane. Rossatto et al, 2014, evaluating the effectiveness of these silanes in the adhesion between fiberglass post and dual resin cement, showed lower values of bond strength of the immediate hydrolysis silane than pre-hydrolyzed silane (20).

The analysis of the failure mode after the micro-shear bond strength test showed a tendency towards greater cohesive failure of the base resin composite when the bond strength values were higher, except for the "as received" of immediate hydrolysis silane. Adhesive failures were observed mainly in groups that had a lower bond strength. This justifies the lower adhesive strength of silanes after one year of use, which weakened the adhesive line between the old resin composite and the new one used to simulate the repair (19).

The CA methodology assesses material wettability and surface energy. That is, the smaller the contact angle, the greater the surface energy (21). In the present study, the silane type influenced the surface energy of the restorative material. However, the storage time did not influence the result. The pre- hydrolyzed silane primer had lower CA than immediate hydrolyzed silane primer. The CA varied according to the chemical changes caused by the application of primer on resin composite surface (22). The higher CA might also be an indicative that silane molecules remain effectively bonded to the hydroxyls on resin composite surface (22-24). After silane application, the energy balance is modified because MPTMS molecules will bond to Si-OH on the surface increasing the CA (23, 24).

The form of pre-hydrolyzed silane storage after first opening did not influence the bond strength of repairs in resin composite. The temperature influences more on the volatilization of the solvent after application on the restorative material than on the stability of the silane inside the bottle. However, the immediate hydrolysis silane showed bond strength similar to the "as received" when stored in a room temperature and

decreased the bond strength in low temperatures. These results can be explained due to the fact that one of the components changes during storage in the low temperatures, leaving the material with a greater amount of bubbles on the sample surface.

The present study showed that the effectiveness of the silane decreases after the silane primer opening even if the product within the validity. Care must be taken not to use the silane long after opening the bottle, except for immediate hydrolysis silane, which when stored at room temperature can maintain their effectiveness in adhesion in resin composite repairs. So, it is suggested to dentists who do not use silane often in clinical practice, that they opt for immediately hydrolyzed silanes and that they be stored at room temperature. This study evaluated only two types of silane in the marked, and a one resin composite, which is one of the limitations of this work. In addition, future studies should be carried out by performing thermocycling after the preparation of the micro-shear specimens and the evaluation of the bond strength with other materials having the silica in the composition, such as ceramics and fiberglass post. In addition, longitudinal clinical studies are needed to evaluate silane performance clinically.

CONCLUSION

The type of silane and the storage influenced the bond strength in resin composite repairs. The pre-hydrolyzed silane initially presented higher bond strength, however, after one year it presented a decrease in adhesion. The immediate hydrolysis silane maintains stability of the adhesion values after one year at room temperature and decrease bond strength in low temperatures.

ACKNOWLEDGMENTS

This study was performed at the Research Center for Biomechanics, Biomaterials and Cell Biology (CPbio/UFU) and financed by the Coordination of Superior Level Staff Improvement – CAPES/ (Finance Code 001). The authors are grateful to Scanning Electron Microscopy Laboratory of the School of Chemical Engineering of Federal University of Uberlandia (FEQUI-UFU) for the SEM analysis and to Mechanical Engineering School.

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TABLES

Table 1- Compositions of the materials use on the composite resin repair

Commercial name	Manufacturer	Material	Composition	Batch
Ambar APS	FGM, Joinville, SC, Brazil	Condition-two step adhesive system	Methacrylic monomers, photoinitiators, co-initiators, stabilizer inert filler (silica nanoparticles), and ethanol	061217
Condac37	FGM, Joinville, SC, Brazil	37% phosphoric acid	37% phosphoric acid, water, pigments and silicon dioxide	120220
Silano	Dentsply Ind. e Com. Ltda., Petrópolis, RJ, Brazil	Silane	Primer: 95% ethyl alcohol and silane A 174. Activator: 95% ethyl alcohol and glacial acetic acid.	359792L
Prosil	FGM, Joinville, SC, Brazil	Silane	3-Methacryloxpropyltrimethoxysilane, ethanol, water	311018
Herculite Precis	Kerr, Orange, CA, USA)	Nanohybrid composite resin	Methacrylate ester monomers, inert mineral fillers, activators, and stabilizers	6350184
Óxido de alumínio	Bio-art, São Carlos, SP, Brazil	Aluminum oxide particles	Aluminum oxide (Al_2O_3)	49641

Table 2. Means (standard deviation) of microshear bond strength data, according the silane type and storage (n=12)

	As receved (AR)	Room temperature (RT)	Low temperature (LT)
Pre-hydrolised (PH)	14.54 (2.53) Aa	9.72 (1.51) Bb	9.5 (0.99) Ba
Immediate hydrolisis(IH)	12.14 (2.09) Ab	13.43 (1.21) Aa	8.21 (1.3) Bb

Distinct letters indicate statistical difference ($p < .05$). Capital letters indicate difference on the storage and lower letters indicate difference on the silane typ

Table 3. Frequency distribution (%) of failure pattern of the groups in the study.

Group	Adhesive failure	Cohesive failure in resin composite base	Cohesive failure in new resin composite	Mixed failures	
IH-AR	62.5%	29.2%	0	8.3%	B
IH-LT	75%	20.8%	0	4.2%	B
IH-RT	22.9%	58.3%	0	18.8%	A
PH-AR	10.4%	87.5%	0	2.1%	A
PH-LT	56.3%	37.5%	0	6.25%	B
PH-RT	50%	50%	0	0	B

Table 4. Means (standard deviation) of contact angle data, according the silane type and storage (n=12)

	As received (AR)	Room temperature (RT)	Low temperature (LT)
Pre-hydrolised (PH)	58 (3.0) A	53 (2.0) A	54 (.0) A
Immediate hydrolisis(IH)	76 (5.0) B	72 (2.0) B	74 (3.0) B

Distinct letters indicate statistical difference ($p < .05$).

Figure 1

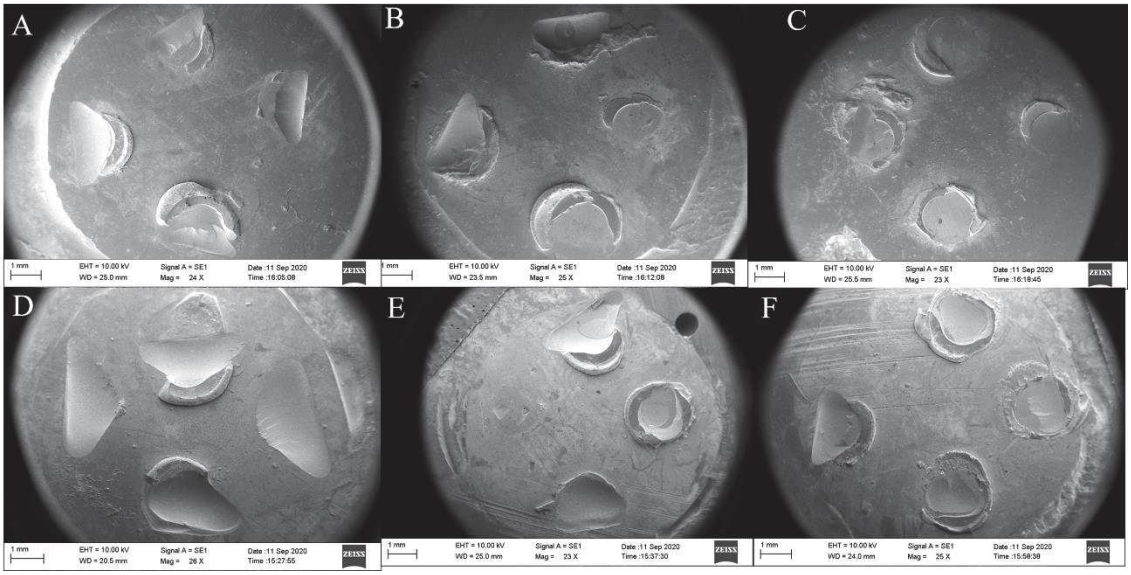

Figure captions:

Figure 1: **(A)** IH-AR, **(B)** IH-RT, **(C)** IH-LT; **(D)** PH-AR, **(E)** PH-RT, (**F)** PH-LT

Capítulo IV

Influence of silane, universal adhesive and hydrolytic stability on the repair bond strength.

Ana Laura Rezende VILELA[a]-

Amanda das Graças SOARES[b]

Alexandre Coelho MACHADO[c]

Marcela Gonçalves BORGES[d]

Murilo de Sousa MENEZES[e]

a- Universidade Federal de Uberlândia (UFU), Departamento de Odontologia, Uberlândia, MG, Brasil; analaurarvilela@gmail.com; https://orcid.org/0000-0002-1020-8273
b- Universidade Federal de Uberlândia (UFU), Departamento de Odontologia, Uberlândia, MG, Brasil; soaresg.amanda@gmail.com; https://orcid.org/0000-0002-8980-8959
c- Universidade Federal de Uberlândia (UFU), Escola Técnica em Saúde, Uberlândia, MG, Brasil; alexandrecoelhomachado@ufu.br; https://orcid.org/0000-0003-0160-5688
d- Universidade Federal de Uberlândia (UFU), Departamento de Odontologia, Uberlândia, MG, Brasil; marcelaborgesufu@yahoo.com.br; https://orcid.org/0000-0003-4000-1916
e- Universidade Federal de Uberlândia (UFU), Departamento de Odontologia, Uberlândia, MG, Brasil; murilomenezes@ufu.br; https://orcid.org/0000-0001-5912-310X

Corresponding author:
Prof. Dr. Murilo de Sousa Menezes
Federal University of Uberlandia, School of Dentistry, Department of Restorative Dentistry and Dental Materials. Avenida Pará, 1720, Bloco 4L, Anexo A, Sala 34, Campus Umuarama Uberlândia - Minas Gerais – Brazil, Tel.: +55 34 3225-8105; Fax.: +55 34 3218 2279 E-mail address: murilomenezes@ufu.br

Influence of silane, universal adhesive and hydrolytic stability on the repair bond strength.

ABSTRACT

Evaluate the bond strength of silane primers and a universal adhesive with and without aging for composite resin repair. One universal adhesive (AU), two silane primers (pre-hydrolyzed (PH) and immediate hydrolysis (IH). Specimens were prepared with composite resin embedded in polystyrene resin and aged a month in distilled water at 37ºC. A half of silane primers and a Universal adhesive were submitted for aging process (AB, 48°C/1month) and the other half were used without aging (NB). The composite resins specimens were sandblasted with aluminum oxide for the surface treatment roughening, cleaned with phosphoric acid and then the silane and adhesive system were applied. The experimental group that received the application of the universal adhesive had not previously applied a silane layer. Four specimens per discs were made with resin composite. The specimens were submitted to the microshear bond strength (μSBS) and failure mode analysis. The statistical analysis was performed by Two-way ANOVA, Tukey's tests and Kruskal Wallis (α = 0.05). The different bonding products do not present difference statistically for "NB". However, on "AB" the "AU" showed the highest bond strength. The "PH" statistically decreased adherence after aging. The failure mode showed more cohesive failure in composite resin base. Therefore, it was concluded the pre-hydrolized silane present more hydrolytic instability than immediate hydrolysis silane and universal adhesive.

Key Words: silanes, adhesion, dental materials, bond strength, composite resins.

INTRODUCTION

The failures frequently reported for posterior restoration are caries, tooth or restoration fractures[1], in anterior restoration the failure is mainly related to esthetics factors[2]. The treatments for these failures range are replacement or repair restoration[3]. The replacement is directly related to loss of tooth structure and this can contribute to the acceleration of the restorative cycle[3-5]. For this reason, many dentists use composite resin repair as an alternative for this type of treatment[6]. This procedure present several advantages such as being a faster procedure with lower cost, patient-friendly[7] and lower chance of pulp iatrogenic[8].

On the composite resin recently confectioned and unreacted monomers are still available for chemical bonding with the fresh composite increment. However, on older composite restoration, the unreacted monomers are leached out, and there is no chemical bond available for bonding with a fresh composite[9, 10]**.** Mechanical interlocking is the most significant factor to increase the bond strength between resin repair and aged composite resin. This step increases the roughness and free surface energy, through removal the superficial layer of resin deteriorated by the oral environment and increases the possibility of the material to offer a greater amount of carbon with the available free linkage[9]. Currently, there are several protocols for repair roughening, such as sandblasted with aluminum oxide[11], diamond burs[12, 13], hydrofluoric acid and phosphoric acid [14, 15].

Based on FDI World Dental Federation, the following treatment steps can be considered as mandatory when performing repairs of partially defective composite restoration[3]. Surface roughening using diamond burs[2], air abrasion or silica coating[1], application of a silane coupling agent or universal primer [4], and application and adhesive[16].

The use of silane in the repair of composite resin restorations is already a consensus in the literature, since the aged composite resin restoration do not have free monomers on the surface capable of bonding to monomers of the new composite resin[9, 10, 17-19.] Silane coupling agent, a monomer composed of reactive

organic radicals and monovalent hydrolysable groups that promote a chemical adhesion between the inorganic phase and organic phase[20, 21]. The most used silane in dentistry is Ƴ- MPS (Ƴ-methacryloyloxypropyltrimethoxysilane), which has a methacrylate group at one end of the molecule[20, 21]. To perform this function, the silane needs undergo to a hydrolysis. This hydrolysis occurs through the manipulation of the silane with a solvent, which in its majority is composed of ethanol or water[20, 21]. The acetic acid present in the silanes has the function of catalyzing the reaction, since the silanes present a faster hydrolysis reaction at acid pH [20, 21]. The hydrolysis of silane occurs to form silanol, which, when bond to silica, forms a cross-linked siloxane bond, generated from the condensation reaction[20, 21]. Silane can be found on the market in several formulations. The bonding agent commonly used are single-bottle silanes, which contain the silane, solvent and catalyst in the same bottle, called pre-hydrolyzed silane[20]. However, studies show that this silane, after its first use, has a short shelf life, as it can generate a condensation reaction of dehydration, inactivating the silanois contained in the bottle[22]. The silanes of two bottle, contain silane and ethanol in one bottle and the acetic acid solution in another, these are mixed minutes before the restoration is carried out.

The most recent formulation is universal adhesives containing silane. Universal adhesives have recently been introduced to the market[10]. The Universal adhesives contains: silane, HEMA, MDP, and Bis-GMA combined into a one-bottle solution. 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) containing adhesive have been shown to provide a reliable bond to indirect restorative materials and tooth substrates[23]. An addition, several manufacturers have added silanes in their formulation to enable their use in repairs and indirect restorations without the need for separate application of silane[24]. However, the literature is scarce in relation to the bond strength of silane in closed bottle, after aging for hydrolytic stability analysis.

Therefore, the objective of this study is evaluate the adhesion performance of composite repairs using closed bottles of pre-hydrolyzed, immediate hydrolysis silanes and universal adhesive containing silane, aged or not. The null hypothesis

of our study was that there is no difference among pre-hydrolyzed, immediate silanes and universal adhesive immediately or after hydrolytic stability analysis

METHODOLOGY

Two silanes coupling agent: pre-hydrolyzed (PH- Prosil, FGM, Joinville-SC, Brazil) and immediate hydrolyzed (IH- Silano, Dentisply, Pensylvania, USA) and one Universal Adhesive (AU- Single Bond Universal, 3M, ESPE, St. Paul, MN, USA), were evaluated in two levels: new bottle (NB) and 30 days after stored at 48ºC (AB)[25]. The systems are evaluated in microshear bond strength and failure mode analysis. The materials used and their chemical compositions are listed in Table 1.

Composite resin specimens

A total of sixty disc-shaped specimens(n=10) of microparticulated composite resin (Palfique LX5, Tokuyama, Japan), shade A2 were prepared (10 mm in diameter and 1mm in thickness) in a teflon cylindrical matrix under a glass plate. The cylindrical mold was covered with mylar strip. After that, a pressure was applied, for 10 seconds, to extrude excess of composite resin and to obtain a smooth and flat surface in each specimen. The specimens were irradiated on both sides for 20 s using light-aticvated polymerization unit (Valo, Ultradent, Indaiatuba, SP, Brazil – 1.000 mW/cm²). After polymerization, the mylar strip and the glass plate on the top of the mold were removed. The specimens were stored in distilled water at 37ºC. After 24 hours, specimens were embedded in polystyrene resin (Aerojet, Santo Amaro, Brazil) and finished with sequential silicon carbide sandpapers, in increasing order of granulation (600, 800, 1200 and 2000) under irrigation (Politriz Universal, Arotec, São Paulo, SP, Brazil). Then, the specimens were cleaned in an ultrasonic with distilled water for 10 min and stored in distilled water at 37 ºC for 6 months. The distilled water of the specimens was changed weekly.

Specimen Preparation

After 6 months, the specimens were randomly divided into six groups according to the type of silane or universal adhesive and aged. The materials used and their chemical compositions are listed in Table 1. The sandblasting was conducted with a sandblaster device (Bio-Art, São Carlos, SP, Brazil) with 50 μm aluminum oxide particles (Al_2O_3). The nozzle was hold perpendicular to the surface for 10s at a distance of 10mm. The surface of all the specimens were then etched with 37% phosphoric acid for 30 seconds, washed with air/water spray for 60 seconds, and dried with a blast of air for 60 seconds[28]. The pre hydrolyzed silane primer, was applied onto the composite resin for 60s, followed by thorough drying using blast of air until complete solvent evaporation. Two layer of bond agent (Âmbar APS, FGM) was applied, air-thinned and light-cured for 20s. The immediate hydrolysis silane was manipulated by mixing the primer and the activator in a bottle and waiting 5 min before application on the composite resin surface. After silano application was waiting 60s followed by thorough drying using blast of air until complete solvent evaporation. A layer of bond agent (Âmbar APS, FGM) was applied, air-thinned and light-cured for 20s. Universal adhesive was applied according the manufacturer: application adhesive to the composite resin surface and rubbed for 20 seconds (active application). Light jet of air was applied over the adhesive, for approximately 5 seconds, aiming at evaporation of the solvent and light-cured for 10s.

For simulate the composite resin repair restorations, four samples per disc of Tygon tubes (TYG-030, Small Parts Inc., Miami Lakes, FL, USA) were used with an internal diameter and height of approximately 0.75mm and 1.50mm, respectively. The Tygon tubes were placed with a minimum distance of 1.5mm between then and over the aged composite resin. The repair resin composite were the same of aged resin composite specimens. The cylinders were photoactivated with light-activated polymerization unit (Valo, Ultradent, Indaiatuba, SP, Brazil – 1.000 mW/cm², according the manufacture) for 20s, and then the molds were cut longitudinally with a scalpel blade and carefully removed by the same operator. The specimens were stored at 37ºC for 24h in distilled water before the test.

Microshear bond strength (μSBS) test

A caliper (Mittutoyo 530312B10, Tokyo, Japan) was used to measure the dimensions of each resin cylinder used to simulate the repairs before the mechanical test. An orthodontic wire with a diameter of 0.2mm (Morelli, Sorocaba, SP, Brazil) was placed perpendicular to the load axis of the resin tubes, which were parallel to the horizontal plane. A 50N load cell was used to apply an increasing parallel force to the adhesive area. Bond strength was tested using a mechanical testing machine (OM100; Odeme, Luzema, SC, Brazil) with a crosshead speed of 1.0 mm/min until specimen fracture occurred. The bond strength (MPa) of each specimen was calculated according to the following formula: T=F/A, where kgf is the force required for failure (N) provided by the machine and πr^2 is the bonded area (mm^2) of the specimens.

Failure mode analysis

The failure area was examined by stereomicroscopy (Mitutoyo, Kawasaki, Japan) at 40x magnification, to assess the failure modes. There was no premature failure. Failure were classificated by three evaluators as adhesive failure, cohesive failure in aged composite resin, cohesive failure in composite resin repair or mixed failures. The adhesive failures occur at the adhesive interface and cohesive when partial fracture occurs on composite resin. The mixed failure occurs in interface and the composite resin.

Statistical analysis

The μSBS were tested for a normal distribution (Shapiro-Wilk) and for equality of variances (Levene test), followed by parametric statistical test. Two-way analysis of variance (ANOVA) was performed for μSBS. Multiple comparisons were made using the Tukey test. The frequency distribution of failure pattern was compared with Kruskal-Wallis test. All the tests employed $\alpha = 0.05$ significance level, and all the analyses were carried out with the statistical package Sigma Plot version 13.1 (Systat Software Inc, San Jose, CA, USA).

RESULTS

Micro-shear bond strength

The mean values and standard deviations of μSBS are described in Table 2, showing statistically significant difference between experimental groups. The statistical showed the interaction between “bonding product” and “bottle aging” (p=0.014). The different silane primers do not present difference statistically on “NB”. However, on “AB” showed highest bond strength for “AU” (14.57 Mpa) than “PH” (12.98 Mpa) and “IH” (12.19 Mpa). The “PH” showed highest bond strength for “NB” than “AB” (p=0.037). The “IH” (p=0.157) and “AU” (p=0.062) not present difference statistically for “NB” and “AB”.

Failure Mode:

The failure mode is described in Table 3. The most prevalent type of failure cohesive in composite resin base (aging composite) and mixed failure is the second more prevalent failure except for IH-AB. Comparing the failure distribution, Kruskal Wallis analysis (Table 3) showed no statistical difference for experimental groups (p=0.843)

DISCUSSION:

The silane primers and universal adhesive are representative of all the products categories currently available. The analysis of the microshear bond strength revealed that the bond strength decreased according to the type of surface treatment and after aging. Therefore, the hypothesis that there is no difference in storage time and surface treatment was rejected.

The Universal adhesive is a Multipurpose adhesive that was developed to be applied to the dental structure with or without prior conditioning[26]. In addition, some manufacturers have added silane in their formulation, with the aim of would simplify the clinical protocol, thereby reducing chair time and operator errors[27]. Previous studies suggested that additional silane pretreatment do not improve the durable of bonding effectiveness of universal adhesive to lithium disilicate ceramic[28, 29]. Due to the Universal adhesive contain many ingredients other than silane, resulting in fewer silane molecules per area in contact with the ceramic surface, in contrast to the silane primer[29]. Also, elimination of solvents and other

byproducts formed during the silane condensation reaction may be hindered through development of a dense polymer network[29]. Furthermore, the silane interaction with -OH and polar-group containing monomers have long been considered as the reason for silanol deactivation[30]. Regarding composite resin repairs, the use of Universal adhesive without the prior application of silane is still controversial. Some articles related the Universal adhesive is efficient for composite resin repair[31], others report lowest bond strength[10, 27]. In the present study demonstrated high bond strength using only Universal Adhesive to composite resin repair.

Scothbond Universal Adhesive contains 10-MDP, a functional monomer can chemically bond to oxide groups such as SiO2, Al2O3, ZrO2 of the composite resin[9]. 10-MDP may also react with zirconia through oxides group present in both the MDP molecule and zirconia surface[32]. Some studies suggest the chemical interaction of SBU with zirconia surface independent of the application of a silane[32, 33].

Considering the Palfique LX5 composite resin contains zirconia fillers, the 10-MDP monomer may help to promote repair bond strength by providing additional chemical bond (27), that justifies the result of the present study. Some studies collaborate with this result (34, 35), which related the multimode adhesive was tested for repairing aged composites resin and CAD/CAM composite resins, which combines methacryloxydecyl phosphate monomers for adhesion to non-glass ceramic substrate and silane for adhesion to glass-ceramic surfaces[35]. This may explain why the new multimode adhesive yielded the highest failure strengths for repair of the aged composites[35]. 10-MDP is a bifunctional molecule and present an amphiphilic structure, with the vinyl and phosphate groups as the hydrophobic and hydrophilic moieties respectively. The viniyl groups may copolymerize with the resin monomer (methacrylate group) of the resin-based material[33, 36]. Furthermore, the 10-MDP make adhesive interface more resistant to biodegradation. Phosphate esters can also bond directly to the surface hydroxyl groups of non-silica-containing ceramics, such as zirconia, and enhance the hydrolytic stability of bonding more than silane coupling agents[31], that justifies better adhesion after aging in the present study[20].

Another bond agent is silane that that contain two different functional groups that may react and connect various inorganic and organic materials[36]. Silane coupling agents promote chemical bonding by forming siloxane bonds between silicate-containing filler particles exposed on the repair surface and the resin matrix of a fresh resin layer[27]. The silanols of silane primer form a direct siloxane bridge with the hydroxyls of the glass surface after silane pretreatment is applied. Thus, a cross-linked siloxane polymolecular layer is produced thereby forming an interpenetrating polymer network with the composite resin[28]. There are in the market many silane formulations, such as pre-hydrolyzed and immediatly hydrolysis.

Pre-hydrolyzed silane is the most silane primer used in dental offices[20]. This primer is found in a single bottle and in hydrolyzed form[20]. However, this type of silane is unstable and has a short half-life. This can be observed in the present study, that pre-hydrolyzed silane after aging for the analysis of hydrolytic stability showed lower adhesion values, even if it was not opened. The mild aging conditions, limited only to a temperature increase, had a detrimental effect on silanol stability and pot life, even in sealed and unopened vials[25]. The aging conditions accelerated hydrolysis, condensation and intermediate reactions, reflecting the sensitivity of pre-hydrolysed silane in storage conditions[25].

The two-bottle silane primers is primer with hydrolysis perform immediately before the application[20]. This primer contain non-hdrolyzed silane are most dissolved in ethanol in one bottle that is necessary be activated and hydrolyzed by manipulated with an aqueous acetic acid solution in the other bottle[20]. The immediately hydrolysis silanes have more stable values than pre-hydrolysates silane. This result can be explained due to the use of silane immediately after the silane mixed, preventing the self-condensation reaction from occurring, due to the quality of the formed siloxane bond is determinate by the concentration of the silane solution and the surface pretreatment protocol (which determines the number of exposed hydroxyl groups)[36].

The analysis of the failure mode after the micro-shear bond strength test showed a tendency towards greater cohesive failure of the base composite resin

when the bond strength values were higher. The cohesive failures within the aged composite resulted in part from the long period of composite aging which favors this type of fracture at the adherent structure[37].

The hydrolytic instability of silane is one of the main problems of this product, that can be observed in bottles that have never been used, and in bottles used inside dental offices. One way to assess this hydrolytic instability was through a methodology used by Dimitriadi M, where silane vials are stored in an oven at 48ºC for 1 month[25, 30].

The clinical significance of this study is that the silane storage time with the bottle closed influences the adhesion to the composite resin restoration. On the other hand, the universal adhesive that containing silane and 10-MDP monomers is a promising product for bonding composite resin repairs to composite resin restauration, which contains silane and zirconia filler particles. However, more studies are still necessary with ceramic and composite resin with other silanes and universal adhesive at different storage times.

CONCLUSION

Within the limits imposed in the experimental design, it is possible to conclude that the immediate hydrolysis silane, pre-hydrolyzed and the universal adhesive showed no difference in bond strength in composite resin repairs when used immediately upon receipt. On the other hand, the universal adhesive showed better bond strength values after aging. The pre-hydrolyzed adhesive showed lower hydrolytic stability after aging.

ACKNOWLEDGMENTS

This study was performed at the Research Center for Biomechanics, Biomaterials and Cell Biology (CPbio/UFU) and financed by the Coordination of Superior Level Staff Improvement – CAPES/ (Finance Code 001).

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TABLE

Table 1- Compositions of the materials use on the composite resin repair

Commercial name	Manufacturer	Material	Composition	Batch
Ambar APS	FGM, Joinville, SC, Brazil	Condition-two step adhesive system	Methacrylic monomers, photoinitiators, co-initiators, stabilizer inert filler (silica nanoparticles), and ethanol	150818
Ultra-Etch	Ultradent, Indaiatuba, SP, Brazil	35% phosphoric acid	35% phosphoric acid, water, pigments, silicon dioxide	BHWGB
Silano	Dentsply Ind. e Com. Ltda., Petrópolis, RJ, Brazil	Silane	Primer: 95% ethyl alcohol and silane A 174. Activator: 95% ethyl alcohol and glacial acetic acid.	31974M 371974M
Prosil	FGM, Joinville, SC, Brazil	Silane	3-Methacryloxpropyltrimethoxysilane, ethanol, water	060420
Single Bond Unviersal	3M ESPE, St. Paul, MN, USA	Universal Adhesive	MDP phosphate monomer, dimethacrylate resins, HEMA, methacrylate modified polyalkenoic acid copolymer, filler, 2ethanol, water, initiators, silane.	2015500264
Palfique Estelite	Tokuyama Dental Corp., Tokyo, Japan	Composite resin	Silica-zirconia filler, Bis-GMA, TEGDMA, photo initiator Filler load: 82 weight%	E716M1
Óxido de alumínio	Bio-art, São Carlos, SP, Brazil	Aluminum oxide particles	Aluminum oxide (Al_2O_3)	49641

Table 2. Means (standard deviation) of microshear bond strength data (Mpa), according the silane type and storage (n=10)

	New Bottle (NB)	Aged Bottle (AB)
Pre-hydrolyzed (PH)	14,39 (1,35) Aa	12,98(1,9) Bb
Immediate hydrolyzed (IH)	13,14 (1,18) Aa	12,19 (1,70) Ba
Universal Adhesive (AU)	13,32 (1,43) Aa	14,57 (1,44) Aa

Distinct letters indicate statistical difference ($p < .05$). Capital letters indicate difference on the "bonding products" and lower letters indicate difference on the "bottle aging".

Table 3. Frequency distribution (%) of failure pattern of the groups in the study.

Group	Adhesive failure	Cohesive failure in composite resin base	Cohesive failure in new composite resin	Mixed failures	
AU-NB	15%	60%	0%	25%	A
AU-AB	8%	72%	0%	20%	A
PH-NB	15%	63%	0%	22%	A
PH-AB	7%	80%	3%	10%	A
IH-NB	0%	97%	0%	3%	A
IH-AB	20%	62%	3%	15%	A

Considerações Finais

3- CONSIDERAÇÕES FINAIS:

Considerando as limitações metodológicas deste estudo, pode-se concluir que:

3.1. A imersão em bebida ácida seguida da escovação imediata com o dentifrício clareador, aumentou a rugosidade da superfície. Os 30 minutos entre a ingestão da bebida ácida e a escovação foram importantes para diminuir o efeito deletério na restauração de resina composta. A microdureza da resina composta não foi influenciada pelo intervalo entre a ingestão da bebida ácida e a escovação

3.2. O diâmetro do fio ortodôntico influenciou nos resultados da resistência de união ao microcisalhamento e a distância entre os corpos não é relevante quando superior a 1,5 mm. Para padronizar, sugere-se o uso de fio ortodôntico de 0,2 mm de diâmetro e no mínimo 1,5 mm de distâncias entre os corpos de prova.

3.3. O tipo de silano e o armazenamento influenciaram na resistência de união em reparos de resina composta. O silano pré-hidrolisado apresenta inicialmente maior resistência de união; no entanto, o silano de hidrólise imediata mantém os valores de adesão após um ano de armazenamento à temperatura ambiente.

3.4. O silano de hidrólise imediata, pré-hidrolisado e o adesivo universal não apresentaram diferença na resistência de união em reparos de resina composta quando utilizado imediatamente ao recebimento. Por outro lado, o adesivo universal apresentou melhores valores de resistência de união após o envelhecimento. O adesivo pré-hidrolisado apresentou menor estabilidade hidrolítica após o envelhecimento.

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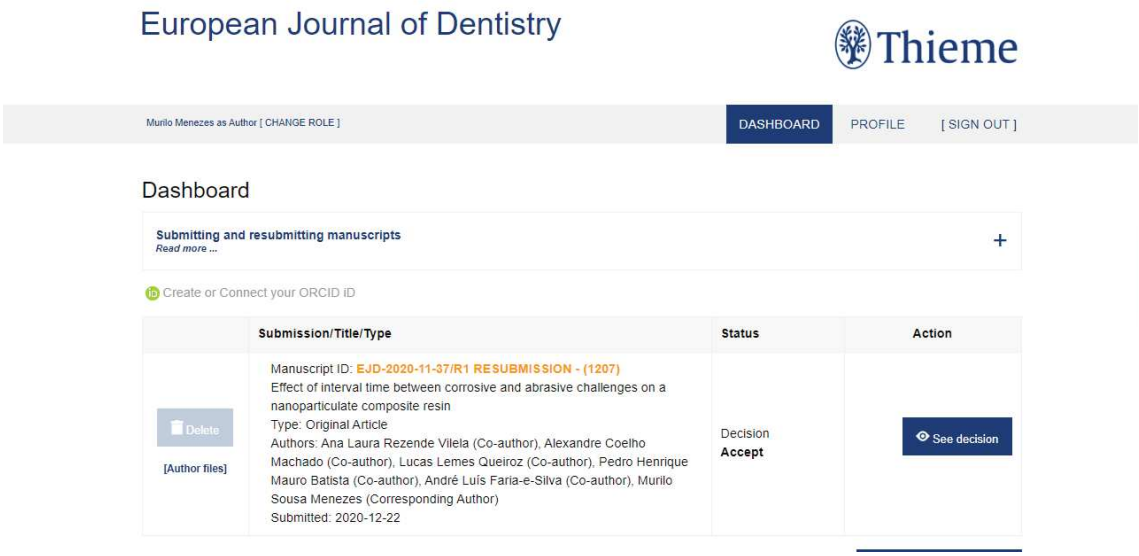
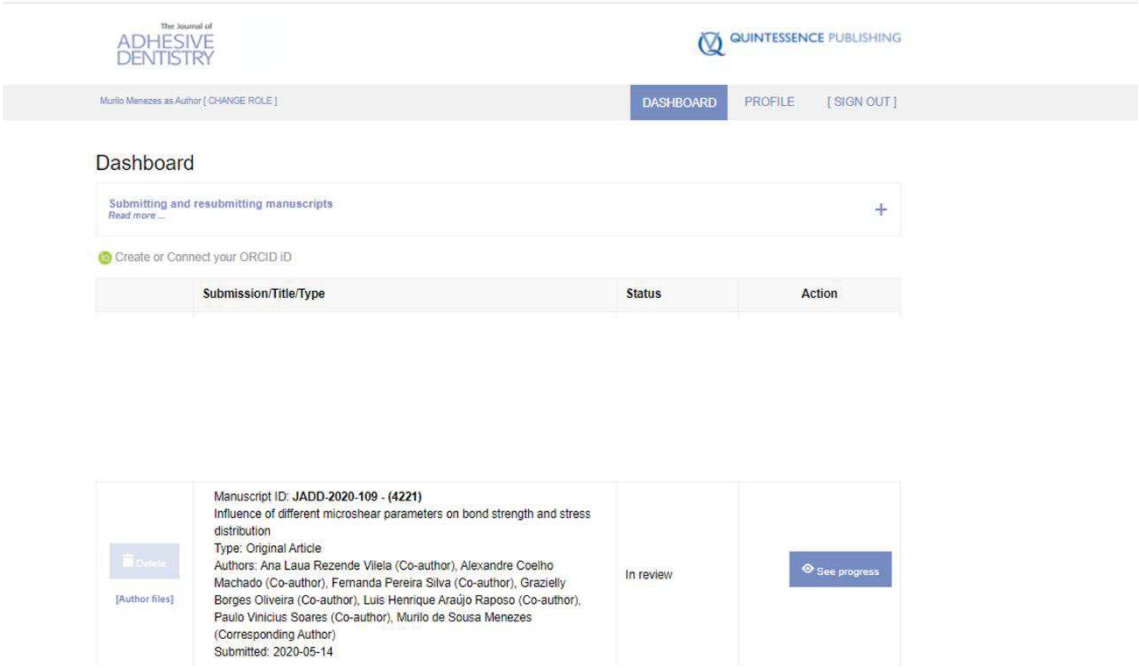
Anexos

1. ARTIGO DO CAPÍTULO 1 ACEITO PARA PUBLICAÇÃO NO PERÍODICO EUROPEAN JOURNAL OF DENTISTRY



1. ARTIGO DO CAPÍTULO II EM REVISÃO NO PERIÓDICO JOURNAL OF ADHESIVE DENTISTRY



The Journal of Adhesive Dentistry

GUIDELINES FOR AUTHORS

The Journal of Adhesive Dentistry is a quarterly 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. Review articles – to topics related to adhesive dentistry.

3a. Invited focus articles – presenting a position or hypothesis on a basic science or clinical subject of relevance 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.

3b. Invited commentaries – critiquing a focus article by addressing the strong and weak points of the focus article 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 a same issue of the Journal.

4. Invited guest editorials – may periodically be solicited by the Editorial Board.

5. Proceedings of symposia, workshops, or conferences - covering topics of relevance to adhesive dentistry related topics.

6. 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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1. Submission via online submission service (www.jad.dk) Manuscript texts should be uploaded as PDF or PC-word files with tables and figures preferably embedded within the PC-word document. A broad range of file formats are acceptable. No paper version required but high resolution photographs or illustrations should be sent to the editorial office (see below).

Online submissions are automatically uploaded into the editorial office's reviewer assignment schedule and are therefore processed immediately upon upload.

2. Submission via e-mail as a PC-word document (pc@quintessence.dk). 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

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Mailing address:

Manuscript Editor

The Journal of Adhesive Dentistry Quintessence Copenhagen European Editorial Office

Fuglevadsvej 27, DK-2800 Lyngby, Denmark

Illustrations that cannot be sent electronically will be scanned at the editorial office so that they can be sent to reviewers via e-mail along with the manuscript to expedite the evaluation process.

Resubmitted manuscripts should also be submitted in the above manner. Please note that supplying electronic versions of your tables and illustrations

upon resubmission will assure a faster publication time if the manuscript is accepted.

Review/editing of manuscripts. Manuscripts will be reviewed by the editor-in-chief, and at least two reviewers with expertise within the scope of the article. The publisher reserves the right to edit accepted manuscripts to fit the space available and to ensure conciseness, clarity, and stylistic consistency, subject to the author's final approval.

Adherence to guidelines. Manuscripts that are not prepared in accordance with these guidelines will be returned to the author before review.

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- 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, title, professional affiliation, 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.

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- Structured abstract. Include a maximum 250-word structured abstract (with headings Purpose, Materials and Methods, Results, Conclusion).

- Introduction. Summarize the rationale and purpose of the study, giving only pertinent references. Clearly state the working hypothesis.

- Materials and Methods. Present materials and methods in sufficient detail to allow confirmation of the observations. Published methods should be referenced and discussed only briefly, unless modifications have been made. Indicate the statistical methods used, if applicable.

- Results. Present results in a logical sequence in the text, tables, and illustrations. Do not repeat in the text all the data in the tables or illustrations; emphasize only important observations.

- Discussion. Emphasize the new and important aspects of the study and the conclusions that follow from them. Do not repeat in detail data or other material given in the Introduction or Results section. Relate observations to other relevant studies and point out the implications of the findings and their limitations.

- Acknowledgments. Acknowledge persons who have made substantive contributions to the study. Specify grant or other financial support, citing the name of the supporting organization and grant number.

- Abbreviations. The full term for which an abbreviation stands should precede its first use in the text unless it is a standard unit of measurement.

- Trade names. Generic terms are to be used whenever possible, but trade names and manufacturer should be included parenthetically at first mention.

REFERENCES

- All references must be cited in the text, numbered according to the alphabetical reference list.

- The reference list should appear at the end of the article, numbered in alphabetical sequence.

- Do not include unpublished data or personal communications in the reference list. Cite such references parenthetically in the text and include a date.

- Avoid using abstracts as references.

- Provide complete information for each reference, including names of all authors (up to six). If the

reference is to part of a book, also include title of the chapter and names of the book's editor(s).

Journal reference style:

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

Book reference style:

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

ILLUSTRATIONS

- All illustrations must be numbered and cited in the text in order of appearance.

Paper version:

- The figure number and first author's last name should be indicated on the back of each photograph or on the mount of each slide. Also indicate the top edge lightly in pencil.

Do not mark author's name on duplicates!

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Radiographs – submit the original radiograph as well as two sets of prints.

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Line drawings – Figures, charts, and graphs should be professionally drawn and lettered large enough to be read after reduction. Good-quality

computer-generated laser prints are acceptable (no photocopies). Lines within graphs should be of a single weight unless special emphasis is needed. Legends – Figure legends should be grouped as a file on a separate sheet and typed double-spaced.

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The Mandatory Submission Form, signed by all authors, must accompany all submitted manuscripts before they can be reviewed for publication.

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- Permission of author and publisher must be obtained for the direct use of material (text, photos, drawings) under copyright that does not belong to the author.
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REPRINTS

The corresponding author is given 50 free reprints of the article. If additional reprints are desired, they must be ordered from the publisher when the page proofs are reviewed by the authors. The publisher does not stock reprints; however, back issues can be purchased.

2. O ARTIGO 3 ENCONTRA-SE NAS NORMAS PARA SER SUBMETIDO PARA O PERIÓDICO BRAZILIAN DENTAL JOURNAL.

Instructions to authors

Scope and policy

The Brazilian Dental Journal is a peer-reviewed (double-blind system) scientific journal that publishes Original Full-Length Papers, Short Communications, Case Reports and Invited Reviews, dealing with the several fields of dentistry or related areas, with open access. Only original papers will be considered for publication. In submitting a manuscript, the authors should state in the cover letter that the material has not been published previously and is not under consideration by another journal in either electronic or printed versions.

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MANUSCRIPTS MUST BE SUBMITTED IN ENGLISH. Authors whose primary language is not English must have their manuscript reviewed by someone proficient in English. **Manuscripts accepted for publication will be submitted to the Technical Review for revision of English grammar and scientific writing and to fit the text into the Journal's standards. The cost of the Technical Review will be charged to the authors. Submission of a manuscript to BDJ implies the acceptance of these terms.** The decision of acceptance for publication relies on the Editors and is based on the recommendation of the Editorial Board and/or *ad hoc* reviewers. Authors of manuscripts not recommended for publication will receive an email explaining the decision. The concepts emitted in the papers published in the BDJ are the sole responsibility of the authors, not necessarily reflecting the Editorial Board's opinion.

All manuscripts will be submitted to peer-review. Authors and referees will be kept anonymous during the review process. Articles accepted for publication become property of the journal.

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The Brazilian Dental Journal in indexed by DOAJ database for public access.

Form and preparation of manuscripts

THE FOLLOWING GUIDELINES MUST BE FOLLOWED CAREFULLY.

General

• The authors must submit the manuscript in Word and in PDF, comprising the title page, text, tables, figure captions and figures (photographs, micrographs, radiographs, schematic drawings, graphs, computer-generated images, etc).

• The manuscript must be typed in Times New Roman 12 font, with 1.5 spacing, 2.5-cm margins at each side. **DO NOT USE** bold letters, watermarks or other resources to make the text visually attractive.

Pages should be numbered consecutively, starting with the summary.

Full-length manuscripts are assembled in the following sections:

1) Title Page
2) Summary and Key Words
3) Introduction; Material and Methods; Results; Discussion
4) Summary in Portuguese (an item necessary for Latin American Indexing Services that will be provided for non-Brazilian authors by the Journal)
5) Acknowledgements (if any)
6) References
7) Tables
8) Figure captions
9) Figures

All titles of sections (Introduction, Material and Methods, etc) must be capitalized in regular font type (not bold).

Results and Discussion **MUST NOT** be joined in a single section.

Short Communications and Case Reports should be divided into appropriate sections.

Products, equipments and materials: the trade name must be followed by the manufacturer's name, city, state and country, within parentheses upon first mention. For further mentions, only the manufacturer's name is required.

All abbreviations must be explained at first mention.

Title page

The first page must contain the title of the manuscript, a short title (maximum of 40 characters, to be used as a running head), author(s) name(s) (no more than 6) and their Department(s), School(s) and/or University (s). **DO NOT INCLUDE** the author's titles (DDS, MSc, PhD, etc.) or position (Professor, Graduate student, etc.).

Provide the name and **complete** address of the corresponding author (inform email, telephone and fax numbers).

The title page must be uploaded at the website as a separate file (not included in the body of the manuscript).

Manuscript

Summary

Introduction

Material and Methods

Results

Discussion

Summary in Portuguese (for Brazilian authors only)

Acknowledgements

References

Journal articles

1. Lea SC, Landini G, Walmsley AD. A novel method for the evaluation of powered toothbrush oscillation characteristics. Am J Dent 2004;17:307-309.

Book

2. Shafer WG, Hine MK, Levy BM. A Textbook of Oral Pathology. 4th ed. Philadelphia: WB Saunders; 1983.

Chapter in a Book

3. Walton RE, Rotstein I. Bleaching discolored teeth: internal and external. In: Principles and Practice of Endodontics. Walton RE (Editor). 2nd ed. Philadelphia: WB Saunders; 1996. p 385-400.

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Figures

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The second page should contain a summary of no more than 250 words, stating the aims, methods, results, and any conclusions drawn from the study. Do not use topics and paragraphs and do not cite references in the Summary.

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Summarize the purpose of the study, giving only pertinent references. Do not review existing literature extensively. State clearly the working hypothesis.

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Present the results in a logical sequence in the text, tables and figures, emphasizing the important information.

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Summarize the findings without repeating in detail the data given in the Results section.

Relate your observations to other relevant studies and point out the implications of the findings and their limitations. Cite pertinent studies.

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References must be numbered consecutively in the text in order of citation, within parentheses, without space between numbers: (1), (3,5,8), (10-15). **DO NOT USE** superscript numbers.

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All authors of each paper should be included in the Reference List unless there are 7 or more. In this case, the first 6 authors should be given, followed by "et al.".

The reference list must be typed at the end of the manuscript in numerical sequence. **No more than 25 references may be cited.**

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Abbreviations of journal titles should conform to those used in Dental Index. The style and punctuation of references must follow the format illustrated below:

Each table with its title must be typed after the text. Tables should be numbered with Arabic numerals. **DO NOT USE** vertical lines, bold letters and capital letters (except the initials).

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Separate parts of composite figures must be labeled with capital letters (A, B, C, etc). Single figures and composite figures must have minimum width of 8 cm and 16 cm, respectively.

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Submission of manuscripts

CHECKLIST FOR AUTHORS PRIOR TO SUBMISSION

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4. In the manuscript, observe:

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- tables, figure captions and figures at the end of the manuscript.

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4. O ARTIGO 4 ENCONTRA-SE NAS NORMAS PARA SER SUBMETIDO PARA O PERIÓDICO BRAZILIAN ORAL RESEARCH.

Instructions to authors

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Brazilian Oral Research - BOR (online version ISSN 1807-3107) is the official publication of the *Sociedade Brasileira de Pesquisa Odontológica* - SBPqO (the Brazilian division of the International Association for Dental Research - IADR). The is rated A2 Qualis Capes (Dentistry), Impact Factor ™ / 20182019 1,508 (Institute for Scientific Information - ISI), is peer-reviewed (double-blind system), and its mission is to disseminate and promote an information interchange concerning the several fields in dentistry research and/or related areas with gold open access.

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The sections of the manuscript must be presented observing the specific characteristics of each type of manuscript: cover sheet (Title Page), introduction, methodology, results, discussion, conclusion, acknowledgments and references.

Title page (compulsory data)

Indication of the themaic area of the research focused on in the manuscript.

Thematic Areas: Anatomy; Basic Implantodontology and Biomaterials; Behavioral Sciences; Biochemistry; Cariology; Community Dental Health; Craniofacial Biology; Dental Materials; Dentistry; Endodontic Therapy; Forensic Dentistry; Geriatric Dentistry; Imaginology; Immunology; Implantodontology – Prosthetics; Implantodontology – Surgical; Infection Control; Microbiology; Mouth and Jaw Surgery; Occlusion; Oral Pathology; Orthodontics; Orthopedics; Pediatric Dentistry; Periodontics; Pharmacology; Physiology; Prosthesis; Pulp Biology; Social/Community Dentistry; Stomatology; Temporomandibular Joint Dysfunction.

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Abstract: Must be presented as a single paragraph (without sub-divisions into sections, containing objective, methodology, results, and conclusions). In the System if applicable, use the Special characters tool for special characters.

Keywords: Ranging from 3 (three) to 5 (five) main descriptors should be provided, chosen from the keywords registered at https://meshb.nlm.nih.gov/search (no synonyms will be accepted).

Introduction: This should present the relevance of the study, and its connection with other published works in the same line of research or field, identifying its limitations and possible biases. The objective of the study should be concisely presented at the end of this section.

Methodology: All the features of the material pertinent to the research subject should be provided (*e.g.*, tissue samples or research subjects). The experimental, analytical, and statistical methods should be described in a concise manner, although in detail, sufficient to allow others to recreate the work. Data from manufacturers or suppliers of products, equipment, or software must be explicit when first mentioned in this section, as follows: manufacturer's name, city, and country. The computer programs and statistical methods must also be specified. Unless the objective of the work is to compare products or specific systems, the trade names of techniques, as well as products, or scientific and clinical equipment should only be cited in the "Methodology" and "Acknowledgments" sections, according to each case. Generic names should be used in the remainder of the manuscript, including the title. Manuscripts containing radiographs, microradiographs, or SEM images, the following information must be included: radiation source, filters, and kV levels used. Manuscripts reporting studies on humans should include proof that the research was ethically conducted according to the Helsinki Declaration (*World Medical Association,* http://www.wma.net/en/30publications/10policies/b3/). The approval protocol number issued by an Institutional Ethics Committee must be cited. Observational studies should follow the STROBE guidelines (http://strobe-statement.org/), and the check

list must be submitted. Clinical Trials must be reported according to the CONSORT Statement standard protocol (http://www.consort-statement.org/); systematic reviews and meta-analysis must follow the PRISMA (http://www.prisma-statement.org/), or Cochrane protocol (http://www.cochrane.org/).

Clinical Trials

Clinical Trials according to the CONSORT guidelines, available at www.consort-statement.org. The clinical trial registration number and the research registration name will be published along with the article.

Manuscripts reporting studies performed on animals must also include proof that the research was conducted in an ethical manner, and the approval protocol number issued by an Institutional Ethics Committee should be cited. In case the research contains a gene registration, before submission, the new gene sequences must be included in a public database, and the access number should be provided to BOR. The authors may use the following databases:

GenBank: http://www.ncbi.nlm.nih.gov/Genbank/submit

EMBL: http://www.ebi.ac.uk/embl/Submission/index.html

DDBJ: http://www.ddbj.nig.ac.jp

Manuscript submissions including microarray data must include the information recommended by the MIAME guidelines (Minimum Information About a Microarray Experiment: http://www.mged.org/index.html) and/or itemize how the experimental details were submitted to a publicly available database, such as:

ArrayExpress: http://www.ebi.ac.uk/arrayexpress/

GEO: http://www.ncbi.nlm.nih.gov/geo/

Results: These should be presented in the same order as the experiment was performed, as described under the “Methodology” section. The most significant results should be described. Text, tables, and figures should not be repetitive. Statistically relevant results should be presented with enclosed corresponding p values.

Tables: must be numbered and cited consecutively in the main text, in Arabic numerals. Tables must be submitted separately from the text in DOC, DOCX, or format (they can be gathered in a single file).

Discussion: This must should discuss the study results in relation to the work hypothesis and relevant literature. It should describe the similarities and differences of the study in relation to similar studies found in literature, and provide explanations for the possible differences found. It must also identify the study’s limitations and make suggestions for future research.

Conclusions: must be presented in a concise manner and be strictly based on the results obtained in the research. Detailing of results, including numerical values, etc., must not be repeated.

Acknowledgments: Contributions by colleagues (technical assistance, critical comments, etc.) must be given, and any bond between authors and companies must be revealed. This section must describe the research funding source(s), including the corresponding process numbers.

References: Only publications from peer-reviewed journals will be accepted as references.

Reference citations must be identified in the text with superscript Arabic numerals. The complete reference list must be presented after the “Acknowledgments” section, and the references must be numbered and presented in Vancouver Style in compliance with the guidelines provided by the International Committee of Medical Journal Editors, as presented in Uniform Requirements for Manuscripts Submitted to Biomedical Journals (http://www.ncbi.nlm.nih.gov/books/NBK7256/). The journal titles should be abbreviated according to the List of Journals Indexed in Index Medicus (http://www.ncbi.nlm.nih.gov/nlmcatalog/journals). The authors shall bear full responsibility for the accuracy of their references.

Spelling of scientific terms: When first mentioned in the main text, scientific names (binomials of microbiological, zoological, and botanical nomenclature) must be written out in full, as well as the names of chemical compounds and elements.

Units of measurement: These must be presented according to the International System of Units (http://www.bipm.org or http://www.inmetro.gov.br/consumidor/unidLegaisMed.asp).

Footnotes on the main text: These must be indicated by asterisks and restricted to the bare minimum.

Figures: Photographs, microradiographs, and radiographs must be at least 10 cm wide, have at least 500 dpi of resolution, and be provided in TIFF format. Charts, drawings, layouts, and other vector illustrations must be provided in a PDF format. All the figures must be submitted individually in separate files (Figure 1a, Figure 1b, Figure 2...) and not inserted into the text file.

Figures must be numbered and consecutively cited in the main text in Arabic numerals. Figure legends should be inserted together at the end of the text, after the references.

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Original Research

Limited to 30,000 characters including spaces (considering the introduction, methodology, results, discussion, conclusion, acknowledgments, tables, references, and figure legends). A maximum of 8 (eight) figures and 40 (forty) references will be accepted. The abstract can contain a maximum of 250 words.

Layout

Title Page
Main text (30,000 characters including spaces)
Abstract: a maximum of 250 words
Keywords: 3 (three)-5 (five) main descriptors
Introduction
Methodology
Results
Discussion
Conclusion
Acknowledgments
References: maximum of 40 references
Figure legends
Figures: a maximum of 8 (eight) figures, as described above
Tables.

Short Communication

Limited to 10,000 characters including spaces (considering the introduction, methodology, results, discussion, conclusion, acknowledgments, tables, references, and figure legends). A maximum of 2 (two) figures and 12 (twelve) references will be allowed. The abstract can contain a maximum of 100 words.

Layout

Title page
Main text (10,000 characters including spaces)
Abstract: a maximum of 100 words
Descriptors: 3 (three)-5 (five) main descriptors
Introduction
Methodology
Results
Discussion
Conclusion
Acknowledgments
References: a maximum of 12 references
Figure legends

Figures: a maximum of 2 (two) figures, as described above

Tables.

Critical Review of Literature

The submission of this type of manuscript will be performed only by invitation of the BOR Publishing Commission. All manuscripts will be submitted to peer-review. This type of manuscript must have a descriptive and discursive content, focusing on a comprehensive presentation and discussion of important and innovative scientific issues, with a limit of 30,000 characters including spaces (considering the introduction, methodology, results, discussion, conclusion, acknowledgments, tables, references, and figure legends). It must include a clear presentation of the scientific object, logical argumentation, a methodological and theoretical critical analysis of the studies, and a summarized conclusion. A maximum of 6 (six) figures and 50 (fifty) references is permitted. The abstract must contain a maximum of 250 words.

Layout

Title page

Main text (30,000 characters including spaces)

Abstract: a maximum of 250 words

Keywords: 3 (three)-5 (five) main descriptors

Introduction

Methodology

Results

Discussion

Conclusion

Acknowledgments

References: maximum of 50 references

Figure legends

Layout

Figures: a maximum of 6 (six) figures, as described above

Tables.

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While summarizing the results of original studies, quantitative or qualitative, this type of manuscript should answer a specific question, with a limit of 30,000 characters, including spaces, and follow the Cochrane format and style (www.cochrane.org). The manuscript must report, in detail, the process of the search and retrieval of the original works, the selection criteria of the studies included in the review, and provide an abstract of the results obtained in the reviewed studies (with or without a meta-analysis approach). There is no limit to the number of references or figures.

Tables and figures, if included, must present the features of the reviewed studies, the compared interventions, and the corresponding results, as well as those studies excluded from the review. Other tables and figures relevant to the review must be presented as previously described. The abstract can contain a maximum of 250 words.

Layout

Title page

Main text (30,000 characters including spaces)

Abstract: a maximum of 250 words

Question formulation

Location of the studies

Critical Evaluation and Data Collection

Data analysis and presentation

Improvement

Review update

References: no limit on the number of references

Layout - Graphic Files

Figures: no limit on the number of figures

Tables.

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Tables, in DOC, DOCX or EXCELL format.

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EXAMPLES OF REFERENCES

Journals

Bhutta ZA, Darmstadt GL, Hasan BS, Haws RA. Community-based interventions for improving perinatal and neonatal health outcomes in developing countries: a review of the evidence. Pediatrics. 2005;115(2 Suppl):519-617. https://doi.org/10.1542/peds.2004-1441

Articles with title and text in a language other than English

Li YJ, He X, Liu LN, Lan YY, Wang AM, Wang YL. [Studies on chemical constituents in herb of Polygonum orientale]. Zhongguo Ahong Yao Za Zhi. 2005 Mar;30(6):444-6. Chinese.

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Pucca Junior GA, Lucena EHG, Cawahisa PT. Financing national policy on oral health in Brazil in the context of the Unified Health System. Braz Oral Res. 2010 Aug;24 Spec Iss 1:26-32.

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Online Books

Foley KM, Gelband H, editors. Improving palliative care for cancer [monograph on the Internet]. Washington: National Academy Press; 2001 [cited 2002 Jul 9]. Available from: http://www.nap.edu/books/0309074029/html/

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Cancer-Pain.org [homepage on the Internet]. New York: Association of Cancer Online Resources, Inc.; c2000 [cited 2002 Jul 9]. Available from: http://www.cancer-pain.org/

Instituto Brasileiro de Geografia e Estatística [homepage]. Brasília (DF): Instituto Brasileiro de Geografia e Estatística; 2010 [cited 2010 Nov 27]. Available from: http://www.ibge.gov.br/home/default.php

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