

MONIZE FERREIRA FIGUEIREDO DE CARVALHO

**TÉCNICA ALCOÓLICA SIMPLIFICADA PARA A CIMENTAÇÃO DE
PINOS INTRARRADICULARES PRÉ-FABRICADOS.**

**FACULDADE DE ODONTOLOGIA
UNIVERSIDADE FEDERAL DE MINAS GERAIS
BELO HORIZONTE**

2018

Monize Ferreira Figueiredo de Carvalho

**TÉCNICA ALCOÓLICA SIMPLIFICADA PARA A CIMENTAÇÃO DE
PINOS INTRARRADICULARES PRÉ-FABRICADOS.**

Tese apresentada ao Programa de Pós-Graduação em
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parcial à obtenção do grau de Doutor em Odontologia -
área de concentração em Clínica Odontológica
Orientador: Prof. Dr. Allyson Nogueira Moreira
Coorientador: Prof(a) Dr(a) Monica Yamauti

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FOLHA DE APROVAÇÃO

**TÉCNICA ALCOÓLICA SIMPLIFICADA PARA A CIMENTAÇÃO DE PINOS
INTRARRADICULARES PRÉ-FABRICADOS.**

MONIZE FERREIRA FIGUEIREDO DE CARVALHO

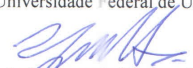
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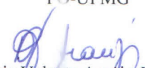
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Prof(a). Allyson Nogueira Moreira - Orientador
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Belo Horizonte, 30 de julho de 2018.



UNIVERSIDADE FEDERAL DE MINAS GERAIS

PROGRAMA DE PÓS-GRADUAÇÃO EM ODONTOLOGIA



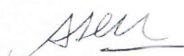
ATA DA DEFESA DE TESE DA ALUNA MONIZE FERREIRA FIGUEIREDO DE CARVALHO

Aos 30 dias de julho de 2018, às 14:00 horas, na sala 3418 da Faculdade de Odontologia da Universidade Federal de Minas Gerais, reuniu-se a Comissão Examinadora composta pelos professores Allyson Nogueira Moreira (Orientador) – FO/UFGM, Vinicius De Magalhães Barros – PUC-MG, Aline Aredes Bicalho – UFU, Luis Fernando Morgan dos Santos Alves – FO/UFGM e Patricia Valente Araújo Jacques Gonçalves – FO/UFGM, para julgamento da tese de Doutorado em Odontologia, área de concentração em Clínica Odontológica, intitulada: **Técnica alcoólica simplificada para a cimentação de pinos intrarradiculares pré-fabricados**. O Presidente da Banca, abriu os trabalhos e apresentou a Comissão Examinadora. Após a exposição oral do trabalho pela aluna e arguição pelos membros da banca, a Comissão Examinadora considerou:

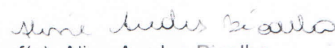
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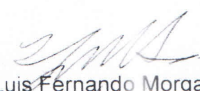
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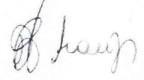
Finalizados os trabalhos, lavrou-se a presente ata que, lida e aprovada, vai assinada por mim e pelos demais membros da Comissão. Belo Horizonte, 30 de julho de 2018.


Prof(a). Allyson Nogueira Moreira


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RESUMO

Pinos de fibra de vidro têm ganhado popularidade devido o módulo de elasticidade próximo ao da dentina, estética e adesão. Contudo, a degradação do sistema adesivo e cimento apresenta-se como uma desvantagem do conjunto pino-cimento-dentina. Assim, a técnica alcoólica foi desenvolvida com a finalidade de minimizar a degradação e aumentar a durabilidade de união. Este estudo *in vitro* teve como objetivo avaliar as propriedades físicas de dois cimentos resinosos e respectiva área de união entre pino e dentina radicular tratada de acordo com dois protocolos de cimentação. Foram utilizados dentes incisivos bovinos. As coroas foram seccionadas na junção cimento-esmalte e as raízes receberam tratamento endodôntico e preparo do conduto. Previamente à cimentação dos pinos de fibra de vidro, as raízes foram divididas aleatoriamente de acordo com o tratamento de superfície radicular: técnica convencional e técnica alcoólica simplificada. Foram empregados dois tipos de cimentos resinosos na fixação do pino: RelyX™ ARC/ Scotchbond Multi-Purpose Plus (3M ESPE) e RelyX™ U200 (3M ESPE). Após a cimentação, as raízes foram armazenadas em 100% de umidade à temperatura ambiente por sete dias para o escaneamento por meio de microtomografia computadorizada (micro CT) ou por 24 horas e 1 ano para o teste de *push-out* e microscopia eletrônica de varredura (MEV). Para análise do micro CT, foi usado o programa NRecon para gerar imagens de reconstrução e o programa CTan para a análise tridimensional das mesmas quanto à porcentagem de porosidades na linha de cimentação. A contração pós-gel (%) dos cimentos foi mensurada empregando-se a técnica de extensometria. Para o ensaio do teste de *push-out*, as raízes foram seccionados e foram obtidos dois espécimes em cada terço radicular. O ensaio usou célula de carga de 5000 N e velocidade de 0,5 mm/min. Os espécimes foram examinados em estereoscópio para identificação do modo de fratura. Imagens foram obtidas por MEV para a avaliação da linha de cimentação. O efeito dos fatores em estudo e sua interação sobre porcentagem de porosidades na linha de cimentação foi verificado por ANOVA e teste *post-hoc de Tukey* ($p \leq 0,05$). A contração pós-gel dos cimentos foi avaliada pelo teste t de *student*. Enquanto a resistência de união foi analisada pelos testes de *Kruskal-Wallis* e *Mann-Whitney* ($p \leq 0,05$). Quando avaliado a porosidade na linha da cimentação a técnica alcoólica com cimento resinoso RelyX™ U200 apresentou os menores valores de porosidade na linha de cimentação para os terços cervical e apical. A contração pós-

gel apresentou valores mais altos para o cimento resinoso RelyX™ ARC. Os valores de resistência de união imediata não diferiram entre os grupos e terços radiculares. Porém, foi afetada negativamente após 12 meses de armazenamento. Apesar do efeito negativo, a técnica alcoólica simplificada associada ao cimento resino RelyX™ U200 apresentou melhor valor de resistência de união quando comparado ao protocolo convencional desse cimento resinoso. Concluiu-se que, o cimento resino RelyX™ U200 com a técnica alcoólica simplificada apresentou a menor porcentagem de porosidade nos terços cervical e apical e que o cimento resinoso RelyX™ U200 apresentou o menor grau de contração de polimerização pós-gel.

Palavras-chaves: Microtomografia por raio-x. Pinos dentários. Cimentos de resina. *Ethanol-wet bonding*.

ABSTRACT

Effect of ethanol-wet bonding on retention of post to root dentin

Fiber post have been an alternative to cast metal posts due to the modulus of elasticity similar to dentin. However, one of the causes of failure is cement degradation. The ethanol-wet bonding was developed with the purpose of reducing this degradation and increasing the durability of bonding between dentin and cement. The aim of this *in vitro* study was to evaluate the mechanical properties of two resin cements and the respective bonding interface between root and dentin treated according to two cementation protocols. Bovine incisor teeth were used. The crowns were sectioned at the cemento-enamel junction and the roots received endodontic treatment and post space preparation. Prior to the cementation of fiberglass, roots were randomly divided according to root surface treatment: conventional technique and ethanol-wet bonding. Two types of resin cements were used to cement the posts: RelyX™ ARC / Scotchbond Multi-Purpose Plus (3M ESPE) and RelyX™ U200 (3M ESPE). After cementation, the roots were stored at 100% humidity at room temperature for seven days for scanning by computerized microtomography (micro CT) or for 24 hours and 1 year for push-out and scanning electron microscopy. For the analysis of micro CT, NRecon software was used to generate tridimensional images and CTan software analyzed the porosity percentage (%) in the luting line. The post-gel contraction (%) of the cements was measured using the strain gauge technique. For push-out test, the roots were sectioned and two specimens were obtained in each root third, with 1.0 mm of thickness each. The test used load cell of 5000N and speed of 0.5mm / min. The specimens were examined in stereoscope to identify the mode of fracture. Images obtained by scanning electron microscopy for the evaluation of the cement line. The effect of the factors under study and their interaction on percentage of porosities in the cementation line was verified by ANOVA and Tukey post-hoc test ($p \leq 0.05$). The post-gel shrinkage was analyzed by Student's t test and bond strength in the root thirds was verified by Kruskal-Wallis e Mann-Whitney test ($p \leq 0.05$). When evaluating the porosity in the cementation line, the ethanol-wet bonding with RelyX™ U200 presented the

lowest values of porosity in the cementation line for the cervical and apical thirds. The RelyX™ U200 resin cement showed the lowest degree of post-gel polymerization shrinkage. Immediate bond strength values did not differ between groups and root thirds. However, storage for 12 months produced a negative effect. Despite this effect, the association between simplified ethanol wet-bonding with RelyX™ U200 exhibit better values of bond strength when compared the conventional protocol for this resin cement. It was concluded that RelyX™ U200 with ethanol wet-bonding had the lowest percentage of porosity in the cervical and apical thirds and RelyX™ U200 resin cement showed the lowest degree of post-gel polymerization shrinkage.

Keywords: X-Ray Microtomography. Dental pin. Resin cement. Ethanol-wet bonding.

LISTA DE ILUSTRAÇÕES

Figura 1: Critérios de exclusão: dentes com ápice radicular aberto e dentes com curvatura radicular significativa, evidenciados pelos círculos....	23
Figura 2: Espécimes seccionados no limite amelocementário.....	24
Figura 3: Exame radiográfico das raízes em duas angulações.....	24
Figura 4: Padronização do comprimento do espécime em 15 mm.....	25
Figura 5: Padronização do diâmetro do espécime com a broca Largo nº3..	25
Figura 6: Preparo do conduto intrarradicular com a broca Largo nº 4.....	26
Figura 7: A: aplicação de peróxido de hidrogênio a 35%. B: aplicação do agente silano no pino.....	27
Figura 8: Tratamento de superfície e controle da umidade realizados no estudo.....	31
Figura 9: A: aplicação de ácido fosfórico 37%.....	31
Figura 10: Aplicação de hipoclorito de sódio a 2,5% previamente à cimentação com RelyX™ U200.....	32
Figura 11: Inserção do cimento resinoso e do pino no conduto e fotoativação.....	32
Figura 12: Micro CT(SkyScan 1272 – Bruker) utilizado para análise do volume de cimento e das porosidades no interior do cimento e da interface dentina/cimento.....	33
Figura 13: inserção da amostra no interior do micro CT.....	33
Figura 14: A: padronização do comprimento digital. B: ranqueamento em amostra de cinza.....	34
Figura 15: Definição digital da área de interesse região cervical (ROI: Região de interesse) sendo a dentina identificada pela estrela, o cimento pelo triângulo e o pino pelo quadrado.....	35
Figura 16: Espécimes obtidos após o seccionamento e identificados conforme a localização, sendo:azul-cervical, vermelho-médio,preto-apical.	36
Figura 17: Aferição da espessura dos espécimes com paquímetro digital.	36
Figura 18: Dispositivo usado na máquina de ensaio mecânico para o teste de <i>push-out</i>	37

Figura 19: Espécime posicionado para teste de <i>push-out</i>	37
Figura 20: A: aparelho de mensuração de contração de polimerização; B: cimento sobre o extensômetro e posicionamento da fonte de luz para ativação do material.....	40

LISTA DE TABELAS

Tabela 1 - Nome, fabricante, composição dos materiais utilizados e instruções para a cimentação dos pinos.....	28
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LISTA DE ABRAVIATURAS E SIGLAS

ARC	Cimento resinoso dual RelyX ARC
Bis-GMA	Bisfenol A-glicidil metacrilato
cm²	Centímetro quadrado
EDTA	Ácido etilenodiamino tetra- acético
Fator C	Fator de configuração cavitária
GPa	Gigapascal
h	Altura
HEMA	Metacrilato de 2-hidroxietilo
Kg	Quilograma
kV	Quilovolt
MEV	Microscopia eletrônica de varredura
micro-CT	Microtomografia computadorizada
ml	Mililitro
mm	Milímetro
Mm/min	Milímetro por minuto
mm²	Milímetros quadrados
MPa	Megapascal
mW/cm²	Megawatt por centímetro quadrado
N	Newton
N/cm²	Newton por centímetro quadrado
NaClO	Hipoclorito de sódio
pH	Potencial hidrogeniônico
r	Raio
rpm	Rotações por minuto
TEGDMA	Trietilenoglicol-dimetacrilato
U200	Cimento resinoso autoadesivo dual RelyX U200
%	Por cento
°C	Grau Celsius
π	PI

SUMÁRIO

1 CONSIDERAÇÕES INICIAIS.....	17
1.1 OBJETIVOS.....	21
1.2 Objetivo geral.....	21
1.3 Objetivos específicos.....	21
2 METODOLOGIA EXPANDIDA.....	22
2.1 Delineamento do estudo.....	22
2.2 Seleção dos dentes.....	23
2.3 Preparo dos dentes.....	23
2.4 Tratamento endodôntico das raízes.....	25
2.5 Preparo radicular para cimentação dos pinos.....	26
2.6 Materiais e cimentação dos pinos intrarradiculares.....	27
2.7 Microtomografia computadorizada (micro CT)	32
2.8 Resistência de união.....	35
2.8.1 Análise do modo de fratura.....	38
2.9 Microscopia Eletrônica de Varredura.....	38
2.10 Contração de polimerização pós-gel dos cimentos resinosos.....	39
2.11 Análise estatística.....	40
3 Effect of ethanol-wet bonding on porosity and retention of post to root dentin: Micro-CT and pushout analyses.....	42
Title page.....	42
Summary.....	43
Introduction.....	44
Material and Methods.....	45
Results.....	49
Discussion.....	50

Conclusion.....	55
Resumo.....	55
Acknowledgements.....	56
References.....	56
Table.....	59
Figures.....	61

Effect of irrigation protocol on bond strength and durability of the adhesion of fiber posts to root dentin.....	62
Title page.....	62
Abstract.....	63
Introduction.....	64
Materials and methods.....	65
Results.....	68
Discussion.....	70
Conclusions.....	73
Acknowledgements.....	73
References.....	73
Table.....	79

4 CONSIDERAÇÕES FINAIS.....	82
REFERÊNCIAS GERAIS.....	83
ANEXO.....	88
PRODUÇÃO CIENTÍFICA (2014-2018)	109

1 CONSIDERAÇÕES INICIAIS

Dentes tratados endodonticamente são estruturalmente diferentes dos dentes vitais pela estrutura dentária remanescente (Gutmann,1992). A perda de cúspides, do teto da câmara pulpar podem resultar na fratura dentária após a restauração final (SIGNORE *et al.*,2009; CECCHIN *et al.*, 2011a). Com isso, o uso de pinos intrarradiculares são geralmente necessários para promover a retenção do material restaurador (TIAN *et al.*, 2012). Dentre os pinos pré-fabricados disponíveis no mercado, os pinos de fibras de vidro ganharam popularidade devido à resistência à flexão e ao módulo de elasticidade (20 GPa) semelhante ao da dentina (18 GPa). Isto possibilita uma distribuição uniforme da tensão e reduz os riscos de fratura das raízes (CECCHIN *et al.*, 2011a; GOMES *et al.*, 2013), além de possibilitar a adesão aos cimentos resinosos (PEREIRA *et al.*,2015) e quando anatomizado, possibilita uma melhor adaptação do mesmo aos canais radiculares (CECCHIN *et al.*, 2011a).

Para que o pino intrarradicular garanta uma boa retenção mecânica e/ou química ao canal radicular, é necessário o uso de sistemas de fixação (CECCHIN *et al.*, 2011b). Diferentes estratégias e materiais têm sido propostos para a correta união entre os pinos de fibra e a dentina radicular: 1) cimento resinoso dual em combinação com o sistema adesivo dual de três passos ou dois passos, 2) cimento resinoso dual em combinação de sistema adesivo autocondicionante e 3) cimentos resinosos autoadesivos (BITTER *et al.*, 2006; MARCHESIA *et al.*, 2012; SARKIS-ONOFRE *et al.*,2014).

Sabe-se que a combinação entre cimento resinoso dual e sistema adesivo de três passos produz resultado confiável na cimentação (CALIXTO *et al.*, 2012). O sistema autoadesivo oferece a vantagem de eliminar o condicionamento ácido prévio, uma vez que contém monômeros multifuncionais de metacrilatos e/ou monômeros ácidos que permitem a adesão sem a desmineralização (MARCHESIA *et al.*, 2012;RODRIGUES *et al.*,2017). Por esse motivo, há uma redução na sensibilidade da técnica (LOGUERCIO *et al.*, 2010) e menor tempo de aplicação (MARCHESIA *et al.*, 2012). Além disso, o resultado da união pino/cimento/dentina radicular tem conferido bons resultados na retenção deste

conjunto (SARKIS - ONOFRE *et al.*, 2014). Nesse contexto, esse tipo de sistema tem ganhado popularidade (FARIA-E-SILVA *et al.*, 2012).

Apesar dos resultados positivos da união mecânica entre pino-cimento-dentina radicular, avaliações clínicas de restaurações com pinos de fibra demonstram que as falhas geralmente ocorrem entre a interface dentina-cimento (MONTICELLI *et al.*, 2003), decorrentes da degradação do cimento (CARRILHO *et al.*, 2005). Alguns mecanismos são responsáveis por essa degradação: o uso de adesivos hidrofílicos que absorvem água (MALACARNE *et al.* 2006), e a degradação das fibrilas de colágeno não encapsuladas presentes na camada híbrida, pelas metaloproteinases de matriz (MMPs) (HOSAKA *et al.*, 2009).

A técnica alcoólica foi desenvolvida com intuito de reduzir essa degradação. Assim, o etanol tem por objetivo deslocar a água presente na dentina, fazendo com que as fibrilas de colágeno fiquem suportadas por esta substância, tornando-as menos hidrofílicas e mais resistentes (PASHLEY *et al.*, 2007; SADEK *et al.*, 2010a). Esta técnica é considerada análoga à técnica seriada de desidratação de etanol para embebição dos tecidos empregada em microscopia eletrônica (NEWMAN & HOBOT, 1999; SADEK *et al.*, 2010b). Porém, a técnica seriada de etanol tem sido pouco atrativa clinicamente devido ao longo tempo de aplicação. Assim, o uso de um protocolo simplificado que garanta a saturação da dentina com etanol absoluto em uma única etapa foi proposto (CECCHIN *et al.*, 2011a; GUIMARÃES *et al.*, 2012)

A técnica alcoólica é recomendada em associação aos sistemas adesivos *etch-and-rise* que podem ser usados previamente à aplicação do cimento resinoso convencional (KIM *et al.*, 2010). Porém, essa mesma técnica pode ser indicada para os cimentos autoadesivos, já que a adesão no canal radicular é um frequente desafio pela geometria dos canais que aprisionam água por tensão superficial, o que torna difícil deslocá-la sem tratamento prévio (PEI *et al.*, 2012).

Com relação a microtomografia computadorizada (micro CT). Este é um método não destrutivo associado à produção de imagens tridimensionais (3D) (UZUN *et al.*, 2015) e que apresenta vantagem quando comparado aos métodos tradicionais na avaliação da interface dentina-cimento-pino (GRANDINI *et al.*, 2005) pela reprodutibilidade e agilidade ao adquirir a imagem, além das amostras poderem ser utilizadas em novos testes (LORENZONI *et al.*, 2013). Permite também a reconstrução e avaliação volumétrica das estruturas internas

e externas (total ou separadamente) superando as limitações dos métodos convencionais (UZUN *et al.*, 2016). Portanto, essa metodologia pode gerar análises importantes na cimentação adesiva de pinos de fibra de vidro.

A associação de metodologias é uma estratégia interessante a fim de melhor explicar os aspectos biomecânicos que envolvem procedimentos restauradores (BRITO-JÚNIOR *et al.*, 2015). Dentre os testes mecânicos existentes para avaliação da resistência de união, o teste de *push-out* apresenta-se como a metodologia mais apropriada para a avaliação de pinos de fibra de vidro ligados à dentina intrarradicular pelo confinamento do compósito dentro das paredes da cavidade preparada, reproduzindo melhor as condições encontradas na clínica (GORACCI *et al.*, 2004; SOARES *et al.*, 2008; BAORO *et al.*, 2014).

A contração de polimerização é resultado de três eventos que ocorrem de forma simultânea: a contração de polimerização dos monômeros, a expansão térmica provocada pela fonte de luz e a expansão térmica provocada pela reação exotérmica do material (SAKAGUCHI *et al.*, 1997). Quando se compara o valor de contração de polimerização dos cimentos resinosos, verifica-se que quanto maior a contração pós-gel, maior a tensão de contração e menor a resistência de união do sistema restaurador (BRAGA *et al.*, 2002; PEREIRA *et al.*, 2015). Entretanto, fatores como contração volumétrica, grau de conversão dos monômeros e fator de configuração da cavidade são importantes para o desenvolvimento da tensão de contração dada pelo material (BRAGA *et al.*, 2005).

A importância da interação entre materiais cimentantes e matriz de dentina desmineralizada deve ser evidenciada afim de auxiliar na durabilidade de união entre dentina-cimento (AYAR, 2016). A técnica alcoólica foi proposta para promover melhor infiltração dos monômeros hidrofílicos e hidrófobos de dimetacrilatos nas matrizes saturadas por etanol com o intuito de promover essa conservação (SAURO *et al.*, 2009; SAURO *et al.*, 2010). Alguns estudos têm examinado o efeito do etanol à dentina radicular sob o efeito de sistemas adesivos (CECCHIN *et al.*, 2011a; EKABARAM *et al.*, 2014). Entretanto, pouco se tem descrito na literatura sobre o efeito da técnica alcoólica simplificada na cimentação de pinos intrarradiculares utilizando cimentos autoadesivos. Por esse motivo, torna-se relevante investigar o efeito sobre a resistência e

durabilidade de união. Além da porcentagem de porosidade na linha de cimentação desse tipo de cimento resinoso.

1.1 OBJETIVOS

1.2 Objetivo geral

Avaliar as propriedades físico-mecânicas de dois cimentos resinosos e respectiva área de união entre pino de fibra de vidro e dentina radicular tratada de acordo com dois protocolos de cimentação após 24 horas e 1 ano.

1.3 Objetivos específicos

- Avaliar a porcentagem total de porosidade na linha de cimentação de pinos de fibra de vidro fixados à dentina radicular (cervical, médio e apical) por meio de dois tipos de agentes cimentantes e distintos protocolos de controle de umidade, usando a metodologia micro CT.
- Avaliar as resistências de união, imediata e tardia, de pinos de fibra de vidro fixados à dentina radicular nos terços cervical, médio e apical, por meio de dois tipos de agentes cimentantes e distintos protocolos de controle de umidade, utilizando o teste de *push-out*.
- Avaliar a área de união, imediata e tardia, de pinos de fibra de vidro fixados à dentina radicular nos terços cervical, médio e apical, por meio de dois tipos de agentes cimentantes e distintos protocolos de controle de umidade, por meio da microscopia eletrônica de varredura.
- Medir o grau de contração pós-gel dos dois cimentos resinosos.

2 METODOLOGIA EXPANDIDA

2.1 Delineamento do estudo

Estudo experimental *in vitro* qualitativo e quantitativo, realizado de acordo com um desenho completamente aleatorizado. As variáveis independentes investigadas foram o tipo de controle da umidade, tipo de cimento resinoso, terço radicular e tempo de armazenamento. Para a primeira variável lançou-se mão de um controle em dois níveis: 1) técnica convencional com controle da umidade radicular com cones de papel absorvente e 2) técnica alcoólica com controle da umidade radicular com etanol absoluto por 60 segundos. Para o segundo, um sistema de cimentação, em dois níveis: 1) cimento resinoso convencional dual RelyX ARC/ Scotchbond Multi-Purpose Plus (3M ESPE, St Paul, MN, EUA); 2) cimento resinoso autoadesivo dual RelyX U200 (3M ESPE, St Paul, MN, EUA). Quanto ao terço radicular, investigou-se as porções cervical, média e apical. Por fim, os tempos de armazenamento analisados foram de 24 horas e 12 meses. As variáveis-resposta analisadas foram: formação de fendas e bolhas na interface cimento-dentina, resistência e durabilidade de união, camada híbrida e grau de contração de polimerização pós-gel.

Para avaliar a interface de união usando o método micro CT, uma amostra de conveniência (n=3), totalizando a amostra de 12 espécimes foi utilizada, devido ao custo e ao tempo de análise.

Para a determinação do tamanho amostral para o teste de resistência de união, utilizou-se o resultado do estudo piloto considerando-se o desvio-padrão, intervalo de confiança de 95% e erro padrão de 5%. Com intuito de compensar eventuais perdas, a amostra foi acrescida de 20%, totalizando 44 raízes dentárias, divididas em quatro grupos representativos (n=11).

Para o teste de microscopia eletrônica de varredura, uma amostra de conveniência de n=2, totalizando-se amostra de 8 espécimes foi utilizada.

Grau de contração de polimerização pós-gel foi baseado em estudo padrão de referência para a técnica utilizada no presente estudo (n=10) (Pereira *et al.*, 2015).

2.2 Seleção dos dentes

O estudo empregou dentes incisivos bovinos, unirradiculares, extraídos após abate do animal no abatedouro Frigorífico Santa Vitória, localizado em Contagem/MG, CNPJ: 01.650.036/0001-83, e doados à pesquisa de acordo com as resoluções do Comitê de Ética Animal (CEUA) da Universidade Federal de Minas Gerais (Anexo I) e armazenados em solução de timol 0,1% a 4° C por até 90 dias. Os critérios de seleção foram: pinças laterais com similaridade anatômica externa e interna (comprimento e diâmetro), ápices completamente formados e sem curvatura radicular. Os critérios de exclusão foram: dentes com curvatura radicular significativa, dentes com fratura radicular e dentes com ápice aberto (Figura 1).

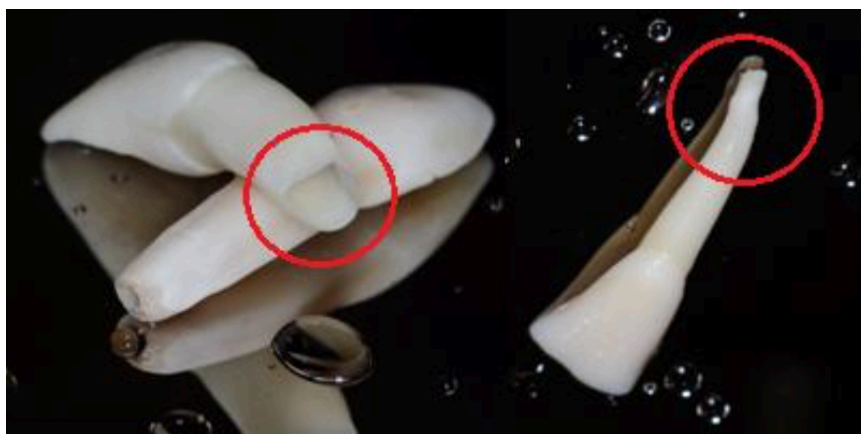


Figura 1: Critérios de exclusão: dentes com ápice radicular aberto e dentes com curvatura radicular significativa, evidenciados pelos círculos.

2.3 Preparo dos dentes

Os dentes foram cortados no limite amelocementário (Figura 2) utilizando um disco diamantado (Discoflex, KG Sorensen, Cotia, São Paulo, Brasil) com auxílio de uma peça reta e armazenados em água destilada refrigerada a 4°C por 96 horas, previamente ao tratamento endodôntico. Em seguida, as raízes foram submetidas a exame radiográfico (Filme radiográfico E-speed, Kodak) em duas angulações a fim de verificar a presença de apenas um canal radicular e a conformação do conduto radicular (Figura 3). Para padronização do comprimento, os espécimes foram seccionados na cortadeira metalográfica de

precisão (Isomet 1000, Buehler, Lake Bluff, EUA) de forma a garantir raízes com comprimento de 15 milímetros (mm) (Figura 4). Para padronização do diâmetro, os espécimes foram selecionados a partir da ponta ativa da broca Largo (Dentsply Maillefer, Ballaigues, Suíça) número 3 (Figura 5). Os condutos que apresentaram resistência para entrada de até 1/3 da ponta ativa da broca Largo foram selecionados e os demais foram excluídos (DALEPRANE *et al.*, 2014; MESQUITA *et al.*, 2013).



Figura 2: Espécimes seccionados no limite amelocementário.

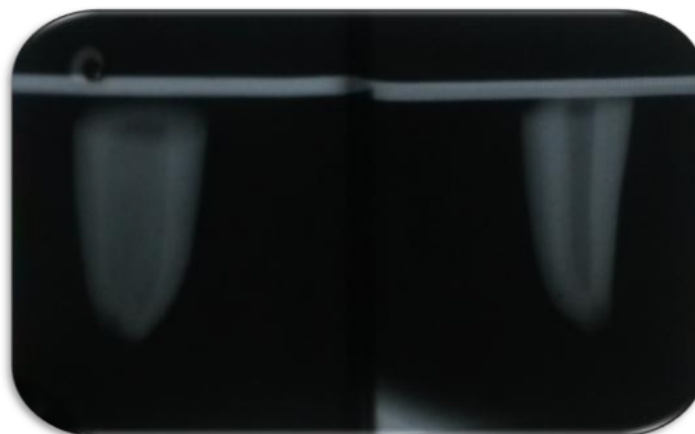


Figura 3: Exame radiográfico das raízes em duas angulações.



Figura 4: Padronização do comprimento do espécime em 15 mm.



Figura 5: Padronização do diâmetro do espécime com a broca Largo n° 3.

2.4 Tratamento endodôntico das raízes

Tecidos pulparem remanescentes foram removidos dos canais radiculares com a lima K-file #10 (Dentisply Maillefer, Ballaigues, Suíça). Os canais foram anatomizados por instrumentos rotatórios de níquel-titânio, Sistema Protaper Universal (Dentisply Maillefer, Ballaigues, Suíça), com o equipamento Xmart com torque a $3,5 \text{ N/cm}^2$ e velocidade de 350 rpm (Dentisply, Petrópolis, Rio de Janeiro, Brasil). Entre cada instrumento utilizado, os canais foram irrigados com de hipoclorito de sódio a 2.5% (Biodinâmica, Ibiporã, Paraná, Brasil) e solução de ácido etilenodiamino tetra-acético (EDTA) (Biodinâmica, Ibiporã, Paraná, Brasil) foi utilizada para irrigação final antes da obturação para limpeza do

conduto. Os canais foram obturados com guta-percha (Odous, Belo Horizonte, Minas Gerais, Brasil) e cimento à base de resina epóxica (AH Plus, Dentsply, Petrópolis, Rio de Janeiro, Brasil), usando a técnica de condensação lateral. Foi realizada uma tomada radiográfica final com o propósito de certificar a qualidade final da obturação. Em seguida, os espécimes foram armazenados em água destilada a 37°C por 7 dias (DALEPRANE *et al.*, 2014; MESQUITA *et al.*, 2014; SILVA *et al.*, 2015).

2.5 Preparo radicular para cimentação dos pinos

Os espécimes foram numerados e identificados e o processo de aleatorização foi realizado por sorteio pelo programa Excel a cada passo e tipo de tratamento. Previamente ao preparo dos condutos, os espécimes foram posicionados perpendicularmente ao solo e fixados em cera utilidade sobre uma placa de vidro para promover apoio durante o manuseio (Figura 6).

Para o preparo do conduto radicular, a guta percha foi removida do interior do conduto mantendo 4 mm de guta percha na região apical, garantindo o selamento da obturação endodôntica e o comprimento de trabalho de 11 mm (ZANATTA *et al.*, 2015). Para o preparo do conduto, utilizou-se brocas largo nº4 para o preparo de no máximo 5 condutos (Dentsply-Maillefer, Ballaigues, Switzerland) para obtenção de 1,3 mm de diâmetro de acordo com as especificações do fabricante, para a cimentação do pino de fibra #2 (Reforpost RX- Ângelus Produtos Odontológicos, Londrina, Paraná, Brasil).



Figura 6: Preparo do conduto intrarradicular com a broca Largo nº 4.

2.6 Materiais e cimentação dos pinos intrarradiculares

Previamente à cimentação, os pinos de fibra de vidro foram tratados com ácido fosfórico à 37% por 1 minutos para limpeza de superfície e posteriormente imersos em peróxido de hidrogênio a 35% (FGM Produtos Odontológicos, Joinville, Santa Catarina, Brasil), por 1 minuto, para exposição das fibras (de Souza Menezes *et al.*, 2011). Em seguida, foram lavados por 1 minuto e secos com jatos de ar (Figura 7 A). Posteriormente, uma camada do agente Silano (Angelus, Londrina, Paraná, Brasil) foi aplicada com auxílio de um microaplicador por 1 minuto. O excesso de silano foi removido com auxílio de jatos de ar por 5 segundos, seguindo as recomendações do fabricante (PEREIRA *et al.*, 2015) (Figura 7 B).

Os materiais usados no estudo se encontram descritos na Tabela 1 e o tratamento da superfície interna radicular e o controle da umidade foram realizados de acordo com os grupos experimentais descritos na Figura 8. Para a fotoativação do sistema adesivo e dos cimentos resinosos, foi utilizado o aparelho de luz halógena de quartzo-tungstênio (OptiLux 501, Demetron Kerr, Orange, Canadá, EUA) por 40 segundos em cada face (coronal, vestibular, lingual) com potência de 600 mW/cm^2 de acordo com o estudo de Pereira *et al.* (2015). No momento da cimentação, as raízes foram cobertas em toda sua lateral com fita isolante preta (Figura 11) a fim de se evitar influência da luz ambiente e garantir maior similaridade com o ambiente bucal (Daleprane *et al.*, 2014).

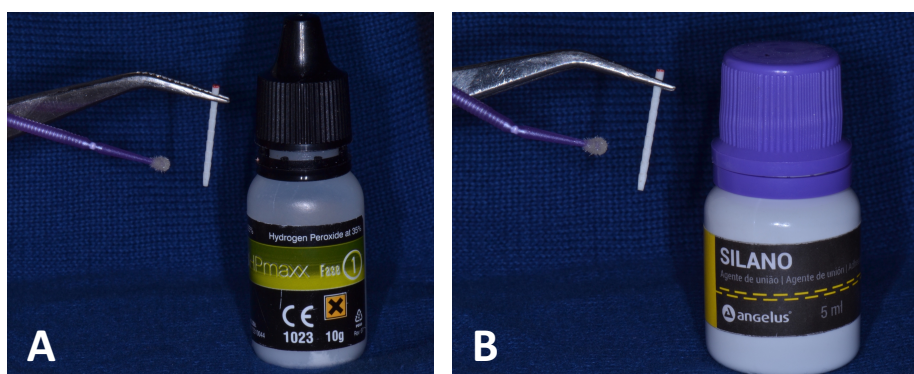


Figura 7: A: aplicação de peróxido de hidrogênio a 35%. B: aplicação do agente silano no pino.

Tabela 1 - Nome, fabricante, composição dos materiais utilizados e instruções para a cimentação dos pinos

Material	Composição	Fabricante	Instruções
Peróxido de Hidrogênio – 35%	Peróxido de Hidrogênio a 35%, Espessante, corante vermelho, glicol e água	FGM Produtos Odontológicos, Joinville, Brasil	Aplicar peróxido de hidrogênio no pino por 60 segundos, lavar rigorosamente por 1 minuto e secar com jato de ar.
Silano	Silano e etanol. Fórmula química: X-R- Si(OR) ₃ n	Angelus, Londrina, Brasil	Aplicar silano no pino por 60 segundos e remover excesso com jato de ar por 5 segundos
Etanol	Álcool absoluto à 99,5%	Itajá, Goiânia, Brasil	Aplicar no conduto radicular por 60 segundos e remover excessos com auxílio de cone de papel absorvente
Ácido fosfórico Condac 37%	ácido fosfórico a 37%, espessante, corante e água deionizada.	FGM Produtos Odontológicos, Joinville, Brasil	Aplicar gel de ácido fosfórico por 15 segundos no interior do conduto radicular. Lavar rigorosamente e remover o excesso de água com cones de papel absorvente (Figura 9).
Adper Scotchbond Multi-Purpose	Primer: solução aquosa de metacrilato de 2-hidroxietilo (HEMA), copolímero ácido polialcenóico, bisfenol A-metacrilato de glicidila (Bis-GMA) Adesivo: bisfenol A-metacrilato	3M, ESPE St. Paul, EUA	Aplicar o Primer e secar suavemente por 5 segundos. Após, aplicar adesivo, remover excessos e

Plus Adhesive	de glicidila (Bis-GMA), metacrilato de 2-hidroxietilo (HEMA)		fotoativar por 10 segundos
Rely X ARC	Pasta A: bisfenol A-metacrilato de glicidila (Bis-GMA), dimetacrilato de trietileno glicol (TEGDMA), Zircônia/Sílica, fotoiniciadores, amina, pigmentos Pasta B: bisfenol A-metacrilato de glicidila (Bis-GMA), dimetacrilato de trietileno glicol (TEGDMA), peróxido de benzoila, Zircônia/Sílica	3M, ESPE St. Paul, EUA	Dispensar o cimento em um bloco de espátula e misturar com espátula durante 10 segundos. Aplicar o cimento no interior do conduto radicular com auxílio da seringa Centrix. Posicionar o pino no conduto radicular e proceder a fotoativação durante 40 segundos em três inclinações para fixação do pino na posição.

Rely X U200	Pasta Base: pó de vidro tratado com silano, ácido 2-propenóico, TEGDMA, fibra de vidro, persulfato de sódio Pasta Catalisadora: pó de vidro tratado com silano, dimetacrilato substituto, sílica tratado com silano, sais de cálcio, hidróxido de cálcio, dióxido de titânio	3M, ESPE St. Paul, EUA	Aplicar hipoclorito de sódio a 2,5% por 60 segundos, lavar com água e secar com cone de papel absorvente, para limpar o conduto, segundo indicação do fabricante (Figura 10). Dispensar o cimento em um bloco de espatulação e misturar com espátula durante 10 segundos. Aplicar o cimento no interior do conduto radicular com auxílio da seringa Centrix. Posicionar o pino no conduto radicular e proceder a fotoativação durante 40 segundos em três inclinações para fixação do pino na posição.
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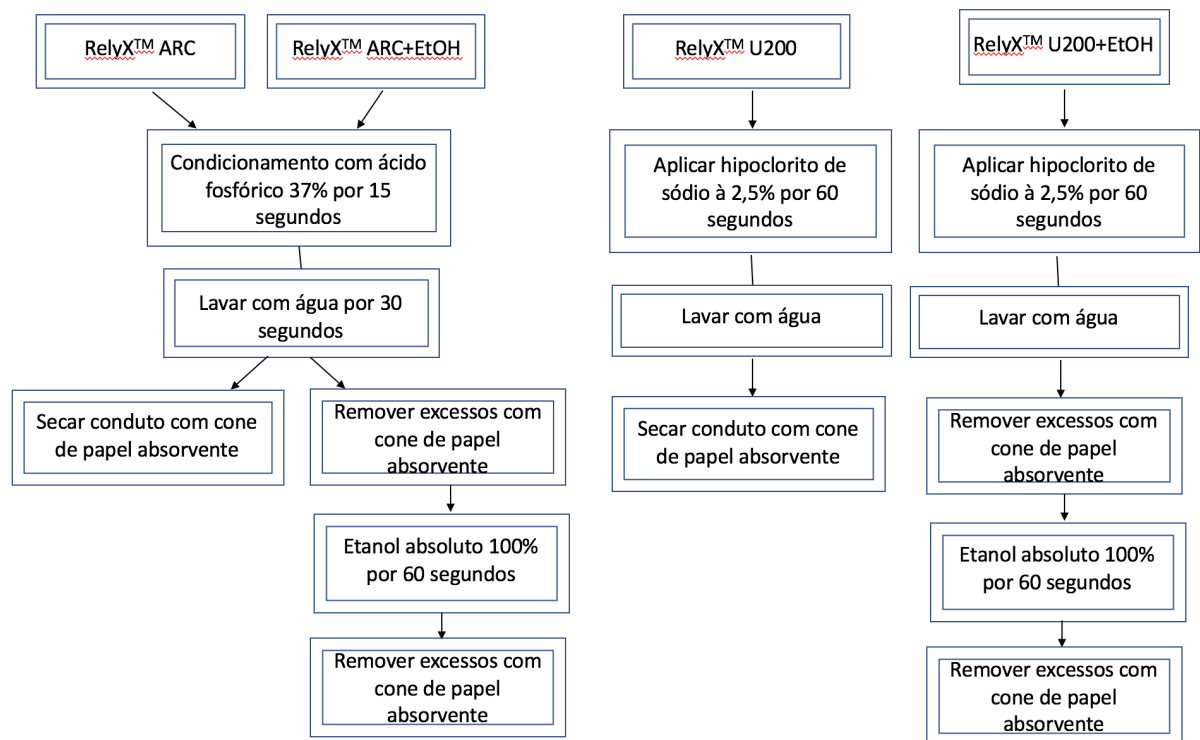


Figura 8: Tratamento de superfície radicular e controle da umidade realizados no estudo.



Figura 9: A: aplicação de ácido fosfórico 37%.

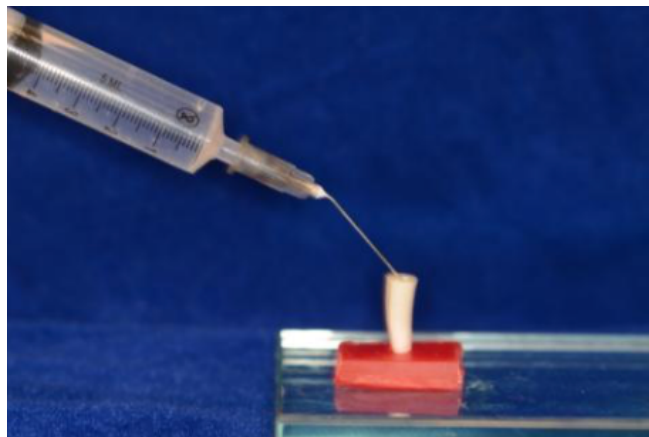


Figura 10: Aplicação de hipoclorito de sódio a 2,5% previamente à cimentação com RelyX™ U200

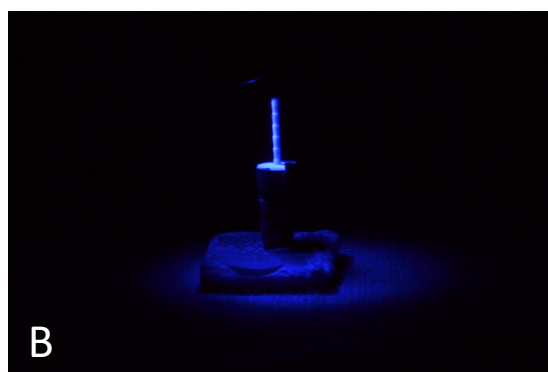
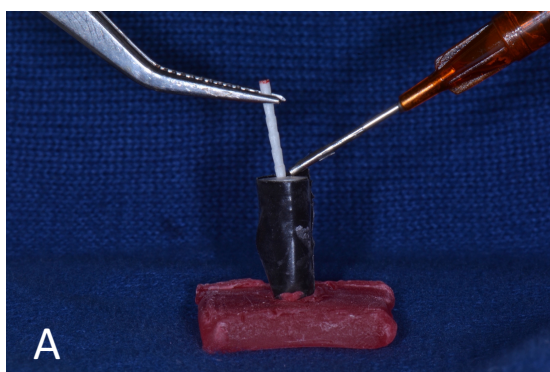


Figura 11 A e B: Inserção do cimento resinoso e do pino no conduto e fotoativação

2.7 Microtomografia computadorizada (micro CT)

O equipamento de micro CT utilizado foi o SkyScan 1272 (Bruker-microCT, Kontich, Bélgica). Trata-se de um sistema compacto de microtomografia composto por uma microfonte de raios-X, um detector de raios-X (câmara), um manipulador de objeto para posicionamento e rotação das amostras e equipamentos eletrônicos associados (Figura 12). A análise por microCT foi realizado no equipamento do Centro de Pesquisa de Biomecânica, Biomateriais e Biologia Celular (CPBio), da Universidade Federal de Uberlândia – UFU.



Figura 12: Micro CT (SkyScan 1272 – Bruker) utilizado para análise do volume de cimento e das porosidades no interior do cimento e da interface dentina/cimento.

As amostras foram inseridas no equipamento de micro CT sempre na mesma posição (Figura 13) e os parâmetros foram estabelecidos para todas as amostras da seguinte forma: filtro de cobre (Cu) de 0,11 mm, resolução em *pixels* de 1224 X 820, voltagem de 100 KV, corrente de 100 μ A e tempo de escaneamento de aproximadamente 30 minutos.

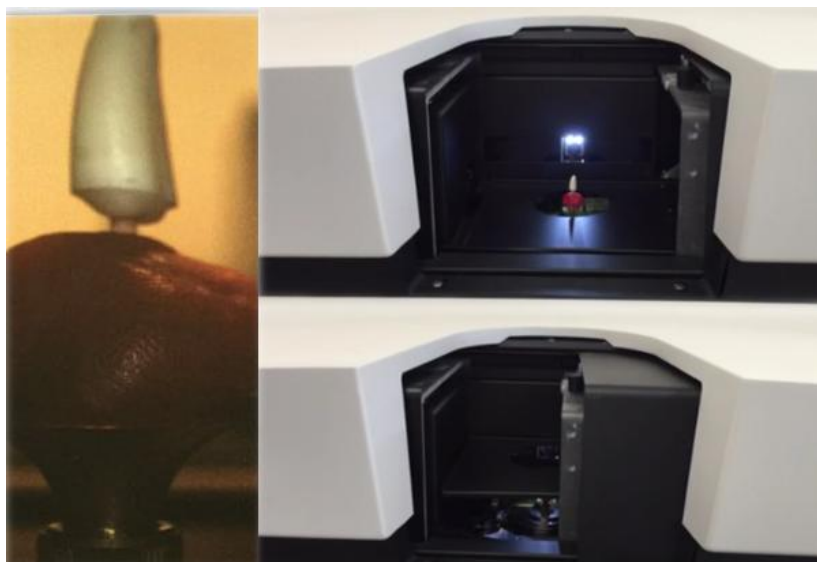


Figura 13: inserção da amostra no interior do micro CT

Após a digitalização, as reconstruções das fatias virtuais das amostras foram realizadas. Foi utilizado o programa NRecon (Bruker-microCT, Kontich, Bélgica) para as reconstruções das imagens de projeção. No momento da

reconstrução, foi padronizado o comprimento digital de todas as amostras. Foram selecionadas 760 fatias entre o topo e a base da amostra a partir do início do pino na porção apical. Em seguida, foi selecionado o mesmo ranqueamento em uma escala de cinza para todas as amostras, padronizadas entre os valores mínimo de 0 e máximo de 0,091893. Nenhuma correção de imagem foi realizada (Figura 13).

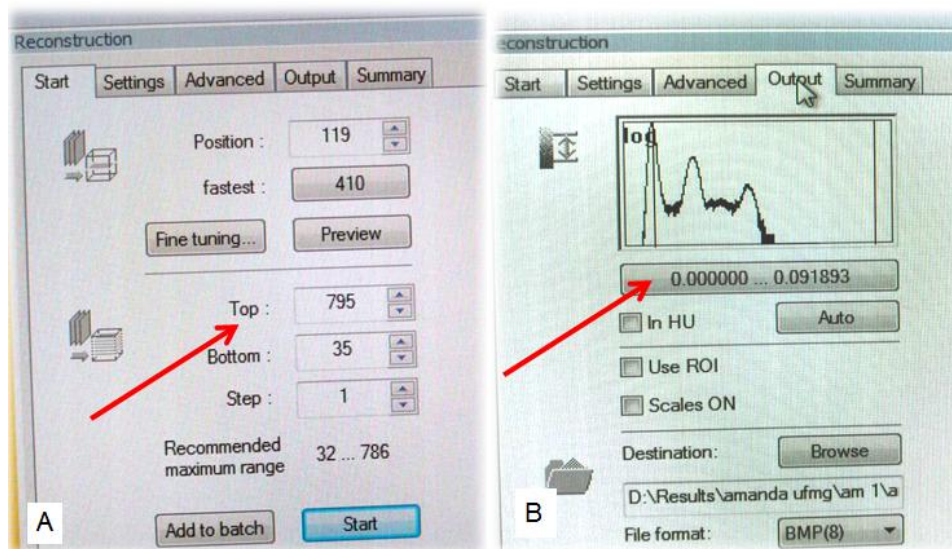


Figura 14: A: padronização do comprimento digital. B: ranqueamento em amostra de cinza

Na sequência, foi utilizado o programa CTAn (Bruker-microCT, Kontich, Bélgica) para a análise dos resultados e renderização de volume. Foi selecionado sempre o mesmo número de fatias conforme a reconstrução. A primeira etapa foi definir o ROI (Região de interesse), que corresponde à área de interesse específica para análise dos resultados. O ROI foi representado por toda a área preparada para a cimentação do pino de fibra de vidro (Figura 15). Após a seleção da área de interesse, foi realizado a binarização das estruturas, sendo marcadas de branco as estruturas analisadas e de preto as estruturas não consideradas na análise.

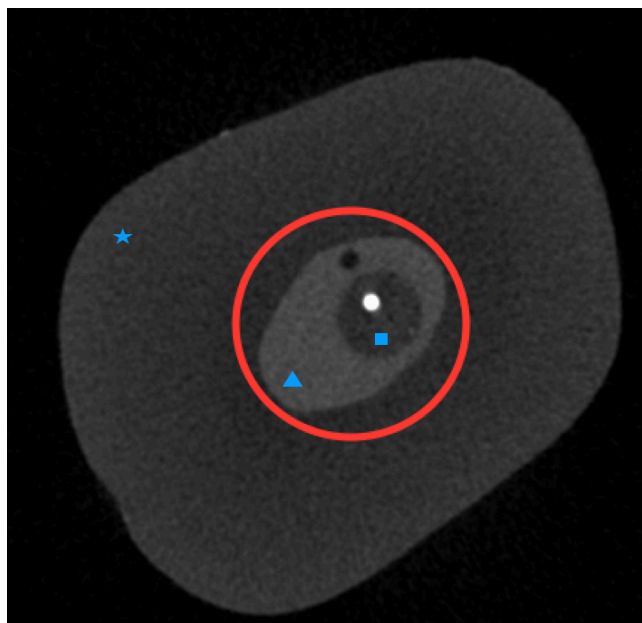


Figura 15: Definição digital da área de interesse região cervical (ROI: Região de interesse) sendo a dentina identificada pela estrela, o cimento pelo triângulo e o pino pelo quadrado.

Logo após, foi solicitado que o programa fizesse a análise morfométrica 3D. Foram obtidos os valores de porcentagem de porosidade na linha de cimentação (em %) (SILVA *et al.*, 2015).

2.8 Resistência de união

Para avaliar a resistência de união, foi realizado o teste de *push-out* em 44 espécimes (n=11) nos tempos: imediato e 12 meses após, para avaliar a longevidade da interface de união. Após o armazenamento em meio úmido a 37° C por 24 horas, as raízes foram seccionadas perpendicularmente ao seu longo eixo, em cortadeira de precisão (Isomet, Büehler, Lake Bluff, IL, EUA), para obtenção de dois espécimes com espessura de aproximadamente 1,00 mm em cada terço radicular (cervical, médio, apical) nos 4 grupos experimentais (Figuras 16). Antes de confeccionar os espécimes foi realizada uma primeira secção na porção mais cervical da raiz com espessura de aproximadamente 0,50 mm e descartada. O objetivo de eliminar essa fatia se dá pelo risco da não polimerização completa do cimento resinoso na presença do oxigênio (DALEPRANE *et al.*, 2016).

Para cada grupo experimental foram obtidas 66 fatias, sendo 33 fatias utilizadas para o teste imediato e a outra metade permaneceu em ambiente úmido para realizar o teste de *push-out* após 12 meses.



Figura 16: Espécimes obtidos após o seccionamento e identificados conforme a localização, sendo: azul – cervical, vermelho – médio, preto – apical.

A espessura dos espécimes foi verificada com um paquímetro digital, com precisão de duas casas decimais (Figura 17). Como preparação para o teste de cisalhamento por extrusão (*push-out*), cada espécime foi posicionado sobre uma base metálica com orifício central de 2,0 mm de diâmetro (Figura 18). Um êmbolo com extremidade de 1,0 mm de diâmetro foi adaptado à máquina de ensaio e posicionado sobre o espécime, de forma a tocar somente o pino, sem entrar em contato com as paredes do conduto radicular (Figura 19).



Figura 17: Aferição da espessura dos espécimes com paquímetro digital.

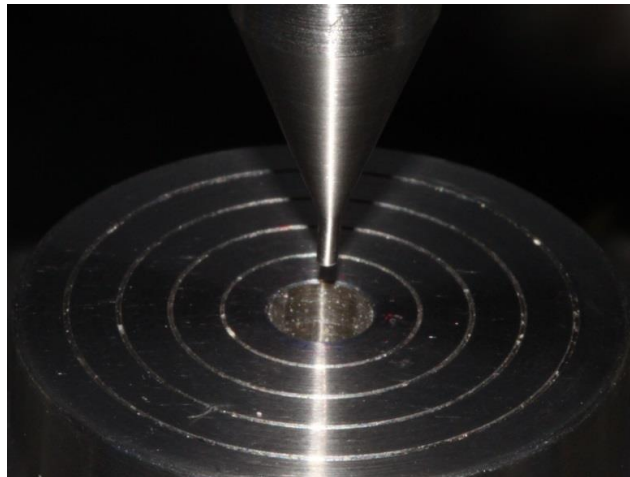


Figura 18: Dispositivo usado na máquina de ensaio mecânico para o teste de *push-out*.



Figura 19: Espécime posicionado para teste de *push-out*.

O ensaio foi realizado em máquina de ensaio universal (EZ-LX Long-Stroke Model, Shimadzu[®], Kyoto, Japão), com uma célula de carga de 5000N, a uma velocidade de 0,5 mm/min no sentido ápice-coronal, até que ocorresse a força máxima para o deslocamento do pino no interior do conduto radicular.

Para obtenção da resistência de união em MPa, a força obtida em Newtons foi dividida pela área da interface de união, que foi calculada pela seguinte fórmula:

$$A = 2\pi rh$$

Onde π é uma constante 3,14; r o raio do pino e a linha de cimento e h a espessura do corte em milímetros.

2.8.1 Análise do modo de fratura

Após o teste de *push-out*, cada espécime foi observado em esteromicroscópio (Zeiss, Jena, Oberkochen, Alemanha), com aumento de 40x para determinar o modo de fratura. Esse foi avaliado de forma independente por dois examinadores calibrados ($\kappa=0,80$) e classificado, conforme se segue: 1) adesiva; 2) coesiva em dentina; 3) coesiva em cimento; 4) fratura mista (ZANATTA *et al.*, 2015). As discordâncias entre os examinadores foram resolvidas por consenso. Os percentuais de fraturas foram registrados.

2.9 Microscopia Eletrônica de Varredura

Dois espécimes representativos de cada condição experimental, resultante da combinação de cada terço radicular (cervical, médio e apical) com cada sistema de cimentação e preparo dentinário, foram selecionados para avaliação da linha de cimentação após 24 horas e 12 meses em microscopia eletrônica de varredura. As secções transversais das raízes foram embutidas em resina ortoftálica (Cristal, Belo Horizonte, MG, Brasil), e depois de 24 horas planificadas com lixas de carbetto de silício de granulações #600 e #1000 (Carborundum Abrasivos, Recife, PE, Brasil) e polidas com lixas de Al₂O₃ de granulações #1200, #2000 e #2500 (Carborundum Abrasivos, Recife, PE, Brasil) em polidora metalográfica (APL-4 Arotec, Cotia, SP, Brasil), sob irrigação com água. Em seguida, a superfície dos espécimes foi desmineralizada com ácido fosfórico 50% por 3 segundos, seguido de enxágue em água corrente por 1 minuto e desproteção por imersão em hipoclorito de sódio a 2,5% por 10 minutos. Logo após, os espécimes foram lavados três vezes com água destilada e imersos em soluções de concentração crescente de etanol (25, 50, 75, 95 e 100%), permanecendo por cerca de 20 minutos em cada concentração. A solução de concentração 100% foi repetida três vezes, com imersão por 10 minutos a cada troca de solução (Daleprane *et al.* 2016).

Em seguida, os espécimes foram mantidos em temperatura ambiente por 10 minutos, e acondicionados em recipiente hermeticamente fechado, contendo sílica gel, por pelo menos 24 horas antes da metalização. Os espécimes foram

metalizados com carbono (sputtering modelo Balzers SCD 050) e observados em microscópio eletrônico de varredura (FEG – FEI, FEG Quanta 200F, Hillsboro, OR, EUA), sob aceleração de voltagem variável entre 15 e 30kV e distância de trabalho variando de 10 a 36 mm. Observou-se nas imagens obtidas a descrição de características morfológicas das linhas de cimentação para cada grupo experimental.

2.10 Contração de polimerização pós-gel dos cimentos resinosos

A análise para contração de polimerização pós-gel dos cimentos resinosos foi realizada no equipamento do Centro de Pesquisa de Biomecânica, Biomateriais e Biologia Celular (CPBio), da Universidade Federal de Uberlândia – UFU.

Amostras dos cimentos manipulados, conforme orientação dos fabricantes (n=10), com volume de 2 mm X 2 mm e 1 mm de espessura foram inseridas sobre o extensômetro bidirecional (CEA-06-032WT-120 M & M, Raleigh, EUA). A fotoativação foi realizada com unidade de luz halógena de quartzo-tungstênio, posicionada com a ponteira distando 1 mm do extensômetro e acionada por 40 segundos, sendo os sinais de intensidade luminosa capturados por um sensor fotocélula (Sanyo AM-1417, Panasonic Ecosolutions Amorton Co., Oregon, EUA) (Figura 20) (SAKAGUCHI *et al.*, 1997; BICALHO *et al.*, 2015; PEREIRA *et al.*, 2015). Para obtenção dos valores de contração volumétrica, foi empregado a fórmula de transformação dos resultados de contração linear em volumétrica, segundo Sakaguchi *et al.*, em 1997, exportada para planilha Excel e apresentada em porcentagem. O medidor de tensão converte alterações de resistência elétrica em alterações de tensão a partir de um circuito fechado e uma resistência interna. Os medidores perpendiculares captam a deformação inicial e final nos eixos X e Y e geram os valores de microdeformação em *microstrain*. A partir da média das microdeformações, obtém-se o valor de variação dimensional em *microstrain*. Os valores são captados durante 10 minutos, incluindo os 40 segundos de fotoativação, evidenciando o valor máximo apresentado. Como se mostra na relação abaixo:

ΔL = (Variação dimensional)

d= (Dimensão linear depois de polimerizado)

D = (Tamanho linear inicial do cimento resinoso)

$$\Delta L = D - d$$

Transformando esta alteração dimensional em volumétrica e percentual temos a seguinte relação:

ΔL = (Variação dimensional linear)

$\Delta L\%$ (Variação dimensional em porcentagem) = $\Delta L \times 100$

ΔV (Contração volumétrica em Percentual) = $\Delta L\% \times 3$

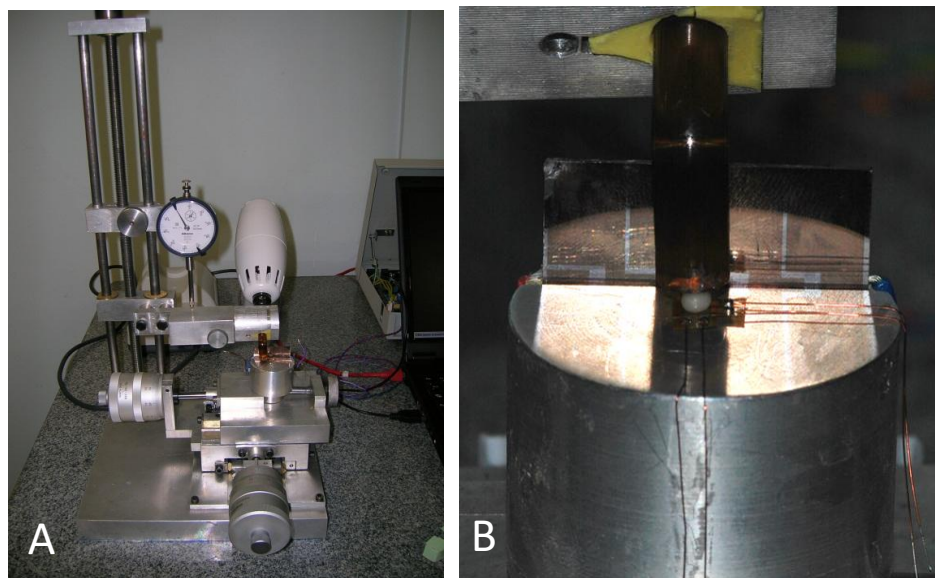


Figura 20: A: aparelho de mensuração de contração de polimerização; B: cimento sobre o extensômetro e posicionamento da fonte de luz para ativação do material.

2.11 Análise estatística

Os dados de porcentagem total de porosidade na linha da cimentação e contração pós-gel apresentaram distribuição normal (Shapiro-Wilk, $p > 0,05$) e equivalência de variância (teste Levene, $p > 0,05$), diferente do observado para resistência de união ($p < 0,05$).

Os dados de porcentagem total de porosidade na linha da cimentação foram analisados por meio do teste de ANOVA seguido pelo teste *post hoc* de Tukey para múltiplas comparações tanto para grupo quanto para terços radiculares.

Para os valores de resistência de união o teste de *Kruskal-Wallis* seguido pelo teste de *Mann-Whitney* para múltiplas comparações foi usado tanto para comparações entre grupos quanto para os terços radiculares.

Os dados de contração pós-gel dos cimentos foram avaliados empregando teste t-Student. O nível de significância adotado foi de 5%. A análise estatística foi realizada utilizando o software SPSS (SPSS Inc., Chicago, IL, USA) versão 21.0.

3 ARTIGOS CIENTÍFICOS

ARTIGO 1

***Title page**

Effect of ethanol-wet bonding on porosity and retention of post to root dentin: Micro-CT and pushout analyses.

Short title: Micro-CT and pushout test for ethanol-wet bonding

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SUMMARY:

This *in vitro* study was to assess the effects of different pretreatments for the retention of fiberglass posts cemented to root canals with different resin luting agents on the percentage of porosity and bond strength. Twelve bovine incisor roots were prepared with largo #3. After post-space preparation, the specimens were randomized into two types of pretreatment groups: water wet bonding and ethanol-wet bonding. Two types of resin cement were used to cement the posts: a dual cure resin cement with 3-step etch-and-rise adhesive system (RelyX™ ARC/ Scotchbond Multipurpose Plus) and a self-adhesive resin cement (RelyX™ U200). After the post cementations, the roots were stored in 100% humidity at room temperature for 7 days. The samples were scanned via computed microtomography (micro-CT). NRecon software was used to generate tridimensional images, and the CTan software was used to analyze the porosity percentage (%) in the luting interface. For the bond strength evaluation, the roots were serially cut, and the slices were submitted to a push-out test. Additionally, the resin cement post-gel shrinkages (%) were measured using the strain-gauge method (n=10). The roots prepared with ethanol-wet bonding with RelyX™ U200 had significantly lower porosity for the coronal and apical thirds ($p<0.05$). The group prepared with ethanol-wet bonding with RelyX™ ARC presented better bond strength results for the coronal and apical thirds ($p<0.05$). RelyX™ ARC (0.97%) produced a higher post-gel shrinkage value than RelyX™ U200 (0.77%). When canals were pretreated with ethanol-wet bonding, there were better outcomes in the percentage of porosity and bond strength.

Keywords: Micro-computed tomography (micro-CT), ethanol wet-bonding, fiber post, resin cement.

INTRODUCTION

The rehabilitation of endodontically treated teeth can be different from vital teeth despite alteration of the coronal tooth structure. Teeth with fewer than two coronal walls usually require specialized additional retention (1). The use of intraradicular posts is necessary to provide retention if the remaining coronal tooth structure is not sufficient (2). Fiber posts present suitable properties to restore endodontically treated teeth. They have high flexural strength and an elasticity modulus (20 GPa) similar to that of dentin (18 GPa) (2), resulting in a lower stress concentration at the root, reducing the risk of root fractures (1). However, these posts failed. The main reason for failure of fiber posts is debonding, with the cement/dentin interface being the most vulnerable link (3). Indeed, some factors can influence the retention of posts in root canals: density and orientation of dentinal tubules (4), root-canal geometry (5), and difficulty in light activating the luting material (3).

The instability of resin-dentin bonds has been attributed to the degradation of hybridized dentin, resin and collagen fibrils. This degradation can occur due to hydrolytic degradation of the resin by water sorption or due to the enzymatic degradation of exposed collagen fibrils (6). For this reason, discovering an alternative that can influence the longevity of postendodontic restorations using fiber posts and increase the longevity of bonding is necessary (7). Ethanol wet-bonding was proposed by Pashley *et al.* (2007). The authors reported the implications of using ethanol to replace water in demineralized collagen matrix, and the infiltration of relatively hydrophobic monomers resins into demineralized dentin created hydrophobic hybrid layers. This technique can prevent water from forming interpeptide H-bonds. Consequently, the dentin matrix can expand with relatively interfibrillar spaces, providing maximal monomer uptake during the infiltration of solvated comonomers. This technique creates hydrophobic hybrid layers that absorb less water and generate durable resin-dentin bonds (8).

Ethanol-wet bonding was initially developed for etch and rinse adhesive systems (8) involving the penetration of resin monomers into a delicate layer of unsupported collagen fibrils exposed to an etching agent (9). This technique was indicated for this type of adhesives to overcome the degradation of non-protected demineralized dentin (5 μm) beneath the hybrid layer (10). In cases of self-etching adhesives, the unsupported collagen

fibrils were thinner (0.5-1.5 μm deep) (8) due to the mechanism of action of ionic resin monomers, which modify and integrate the hybrid layer simultaneously (10).

According to bonding strategies, resin cements are classified in two categories: dual cure resin cement or self-adhesive. Dual-cure resin cements are characterized by the use of a preliminary bonding agent: etch-and-rinse, self-etching or universal adhesives systems (11). Comparatively, the multi-step application technique is time consuming and rather technique sensitive. In brief, self-adhesive resin cements simplified the indirect luting technique by eliminating any pretreatment of the dentin surface (11). The composition of this material was based on acid monomers, which were responsible for demineralizing and infiltrating the substrate, creating micromechanical retention and chemical bonding to hydroxyapatite (3). However, the hybrid layer presents scarce penetration of the cement into radicular dentinal tubules (6). Nevertheless, there is scarce literature on the efficacy of self-adhesive resin cements bonding to root canal dentin concerning ethanol wet-bonding (6). Obtaining a durable resin-dentin interface is crucial for the rehabilitation of endodontically treated teeth using fiber posts. The simplified ethanol wet-bonding technique revealed promising results with respect to fiber post bond strength stability inside the root canal (7).

The aim of this *in vitro* study was to evaluate the adaptation of fiberglass posts cemented to root canals by two different pretreatments regarding the percentages of the total porosity on the interfaces (%), bond strength (MPa) and post-gel shrinkages value (%) of the tested resin cements. The null hypotheses were: 1) the total porosity percentages would not affect the different pretreatments in different portions of the root; 2) the bond strength was not affected by the different protocols in different portions of the root; and 3) the resin cements had similar post-gel shrinkage values.

MATERIAL AND METHODS

SAMPLE SELECTION AND ROOT CANAL PREPARATION

A total of 12 freshly extracted bovine incisor teeth with homogeneous shapes and sizes were collected according to the Animal Ethics Committee of the Institution (protocol #31/2017). After extraction, the teeth were stored in 0.1% thymol solution for up to three months. The bovine incisor teeth showing external and internal anatomical similarity (length and diameter), with a completely formed dental apex and without root curvature, were selected among middle first teeth. Bovine teeth with significant root curvature and/or with root fracture and/or teeth with open apex were excluded from the

study. The crowns were removed using a diamond saw on a precision cutting machine (Isomet 1000, Buehler, Lake Bluff, USA) under constant water irrigation. The obtained roots were 15 mm.

To standardize the canal spaces the sectioned teeth were radiographed, and roots with similar canal diameters were selected. To ensure that similar canal diameters were indeed selected, the canal aperture was tested with a Largo #3 burr (Dentsply, Petrópolis, RJ, Brazil). Only roots that showed some resistance to the Largo #3 drill penetration were used. One trained operator prepared the root canals. The canals were endodontically prepared using rotary instruments (Xmart, Dentsply Maillefer, Ballaigues, Switzerland) under irrigation with 2.5% sodium hypochlorite (Asfer Indústria Química Ltda, São Caetano do Sul, SP, Brazil). Final irrigation was performed with trisodium ethylenediamine tetra-acetic acid (EDTA; Biodinâmica, Ipirorã, PR, Brazil) for 3 minutes, followed by rinsing with water and drying with absorbent paper points. The roots were obturated using a gutta-percha thermoplastic technique with lateral and vertical condensation and AH Plus cement (Dentsply Maillefer) and were then stored in 100% humidity at 37°C for 7 days (12).

POST SPACE PREPARATION AND POST CEMENTATION

The root canals were prepared using Largo burs #3 and #4 (Maillefer-Dentsply, Petrópolis, RJ, Brazil) with a slow rotary speed and 11 mm of length, leaving 4 mm of the apical canal filled. The glass fiber post used was Reforpost RX #2 (Ângelus, Londrina, PR, Brazil). The teeth were randomly divided into four groups (n=3) according to the protocol system used: water-wet bonding with dual cure resin cement (WBD); ethanol-wet bonding with dual cure resin cement (EBD); water-wet bonding with self-adhesive resin cement (WBS); and ethanol-wet bonding with self-adhesive resin cement (EBS). Randomization was achieved using random number sequences generated by the Microsoft Excel RAND function (Office Excel software; Microsoft Corp).

The fiber posts were immersed in 35% hydrogen peroxide (FGM Produtos Odontológicos, Joinville, SC, Brazil) for one minute, rinsed with water for one minute and dried with compressed air (13). This process was followed by the application of a silane coupling agent for one minute (Silano, Angelus, Londrina, PR, Brazil) (5). All of the cement materials were light-activated for 40 seconds in the buccal, lingual and incisal angulations using a quartz-tungsten halogen light curer (600 mW/cm², Demetron 501; Kerr Corporation, Orange, CA, USA) (12). The roots were covered with black tape at the

time of cementation to prevent the influence of ambient light and to create a similarity with the oral environment.

For water-wet bonding groups, the root canals were rinsed with distilled water after etching with 37% phosphoric acid (FGM Produtos Odontológicos, Joinville, SC, Brazil) for 15 seconds (WBD) or 2.5% sodium hypochlorite (WBS) for 60 seconds; excess was removed from the post space using absorbent paper points. With ethanol-wet bonding, the root canal was rinsed with distilled water after etching, excess water was removed with absorbent paper, and the root canals were completely filled with 100% ethanol for 60 seconds (EBD), or after rinsing with water after the application of 2.5% sodium hypochlorite (EBS) and drying with absorbent paper, the root canals were completely filled with 100% ethanol for 60 seconds; subsequently, the excess ethanol was gently removed (2).

After root dentin treatment, two cementation systems were evaluated (Table 1): an etch-and-rise 3-step adhesive system (Scotchbond Multipurpose Plus; 3M ESPE) with a dual cure resin cement (RelyX ARC™, 3M ESPE); and a self-adhesive resin cement (RelyX™U200, 3M ESPE). The bonding agents and resin cements were used according to the manufacturer's instructions (Table 1). After cementation procedures, the roots were stored in water at room temperature for seven days. During this period, the roots were submitted to computed microtomography scanning and push-out testing.

MICRO-CT ANALYSIS

After post cementation, the roots were mounted on a custom attachment and scanned in a micro-CT scanner (SkyScan 1272; Bruker-micro-CT, Kontich, Belgium). The scanner operated at 100 kV and 100 mA using a Cu filter of 0.11 mm. The resolution used was 1224 X 820 pixels - 10 µm. The scanning was performed by 180° rotation around the vertical axis with a rotation step of 0.6. The images of each root were reconstructed using NRecon software, version 1.6.3 (Bruker, micro-CT), to provide axial cross-sections of their inner structures, and the images were reconstructed with standardized digital lengths. CTan software, version 1.12.0 (Bruker-micro-CT), was used for the 3-dimensional analyses. The software analyzed the region of interest (prepared area for post cementation and the interface of cementation) to obtain the results and then analyzed the 3-dimensional morphometry to provide the percentage of the total porosity on the interfaces (%) present in the resin cement layer as well as the interface with the root canal (5).

PUSH-OUT TESTS

Bovine roots (n=3) were fixed in 20 mm X 20 mm acrylic plates with sticky wax and were sectioned perpendicular to the fiber long axis of the post into six slices for each root (approximate thickness of 1.0 mm) using a precision cutting machine under water cooling (Isomet 1.000 Precision Saw, Buehler), obtaining two slices for each third. Each slice was marked on its apical side with an indelible marker, and the thickness was measured using digital calipers (Mitutoyo Digimatic Caliper Serie 500, Mitutoyo Sul Americana, Suzano, SP, Brazil). The specimens were subjected to the push-out bond test with a 5000 N load cell, using a 1-mm-diameter plunger, and the load was applied at a cross-head speed of 0.5 mm/min in the apical-coronal direction using a universal testing machine (EZ-LX Long-Stroke Model, Kyoto, Japan). The plunger was centralized to avoid contact with the dentin. The bond strength was calculated in megapascals (MPa) by dividing the load recorded in newtons (N) by the area (mm²) of the post/dentin interface (2,12).

The failure mode was observed using a stereomicroscope (Carl Zeiss AG) at 40X magnification. The failure mode was assessed by two calibrated operators, and each sample was categorized as follows: adhesive failure, mixed, and cohesive failure (within the cement or dentin) (2,12).

POST-GEL SHRINKAGE MEASUREMENT

The post-gel linear shrinkage was determined using the strain gauge method (n=10) (14). Both resin cements were manipulated and placed on top of a biaxial strain gauge (CEA-06-032WT-120, Measurements Group, Raleigh, NC, USA) that measured the strains during polymerization in two perpendicular directions. The perpendicular strains were averaged because the material properties were homogeneous and isotropic on the macro scale. Each resin cement was light activated using the same LCU, which was used for sample preparation with the light tip placed 1 mm from the surface of the cement. The radiant exposure was 600 mW/cm² and was applied for 40 seconds. A strain conditioner (2101A Series, Micro Measurements Group) converted electrical resistance changes in the strain gauge into voltage changes through a quarter-bridge circuit with an internal reference resistance. The post-gel strains that resulted from polymerization shrinkage were monitored for 10 minutes starting at the beginning of photoactivation (14). The shrinkage strain, which is the linear post-gel shrinkage, of each sample (n=10)

was converted into a percentage and multiplied by three to obtain the volumetric shrinkage.

STATISTICAL ANALYSIS

The interface data, push-out test and post-gel shrinkage values were tested for normal distributions (Shapiro-Wilk, $p < 0.05$) and equalities of variance (Levene's test, $p < 0.05$). The percentage of total porosity and push-out test values were analyzed using two-way ANOVA and Tukey's post hoc test with the type of pretreatment (water wet-bonding and ethanol-wet bonding) and root thirds (coronal, middle, apical) as the factors. The post-gel shrinkage was evaluated using Student's t test, and the data regarding interface integrity were analyzed using two-way ANOVA. Multiple comparisons were performed using Tukey test. Pearson's correlation test was performed to analyze the relationship between the push-out bond strength and the void volume. All of the statistical analyses were performed using SPSS software, version 21.0 (SPSS, Chicago, IL, USA), with a significance level of $\alpha = 0.05$ used in all of the tests.

RESULTS

PERCENTAGE OF TOTAL POROSITY ON THE INTERFACE (%)

The mean volumes and standard deviations of the percentages of the total porosity are presented in Table 2. Results of two-way ANOVA showed that a difference was observed for the factor of pretreatment ($p < 0.001$); however, no significant difference was observed for the post space region ($p = 0.968$). The interaction between the factors of pretreatment and post space region ($p = 0.209$) was not significant. Tukey's test revealed that the percentage of total porosity was significantly lower for EBS for the coronal and apical thirds. The WBD group exhibited a higher percentage of total porosity for the coronal, middle and apical thirds.

PUSH-OUT TESTS

The means and standard deviations of the push-out bond values for all of the groups are presented in Table 3. Results of two-way ANOVA showed that a difference was observed for the factor of pretreatment ($p < 0.001$); however, a significant interaction effect was observed ($p = 0.001$). The root canals prepared with EBD presented the best bonding performance for the coronal and apical thirds. The failure mode distribution is

shown on Figure 1. Adhesive dentin/resin cement interface failure was the most frequent type of failure, irrespective of root dentin third.

There were no significant correlations between the push-out bond strength and the volumes of voids ($p>0.05$).

POST-GEL SHRINKAGE VALUES OF RESIN CEMENTS

The post-gel shrinkage values of the resin cements are presented in Table 4. Student's *t* test revealed that the post-gel shrinkage of RelyX™ ARC differed significantly from that of RelyX™ U200 ($p<0.001$).

DISCUSSION

A hypothetical model was used to investigate the effects of pretreatment of the root canal on the percentages of total porosity on the interfaces and the bond strength of posts along the canal, in addition to post-gel shrinkage. The use of ethanol wet-bonding allowed us to observe a better result for the volume of bubbles and bond strength. The null hypothesis was partially rejected because the pretreatment used in the fixation of fiber posts in the root canals influenced the total porosity and bond strength, and the resin cement type influenced the degree of post-gel shrinkage. However, the root canal factor was not influenced by the region.

Micro-CT is a high-resolution method able to measure in three dimensions (3D) the volumes of gaps and voids around the interface (15). Micro-CT systems using microfocal spot X-ray sources and high-resolution detectors allow projections to rotate through multiple viewing directions to produce 3D reconstructed images of samples (5). Furthermore, they offer the advantage of being a nondestructive method able to analyze all specimens (16), and samples are available for additional bonding testing (5). Evaluating the volume of voids in the cementation of fiber posts is of interest for clinical situations because the presence of voids can affect the longevity of adhesive bonding in three ways: voids located at the interface between the dentin and cement can decrease the contact bonded area; the presence of voids can decrease the strength of the cement and create sites for crack initiation and propagation (16); and the presence of bubbles acts as stress concentration factor, reducing the bond area and reflecting the higher peak of stress on the remaining intact interface (5). In this study, micro CT identified lower porosity on the interface to ethanol-wet bonding with RelyX™ U200. The water wet-bonding obtained high values of porosity, independent of the type of resin cement. These results

could be explained by the miscibility of different solutions; the compatibility between hydrophobic resin and water causing the two liquids to become immiscible due the high value of total cohesive forces, which were different when water was replaced with 100% ethanol (10). When comparing RelyX™ U200 and RelyX™ ARC with ethanol-wet bonding, the lower porosity of RelyX™ U200 might be explained by the lower degree of post-gel shrinkage (1). These findings suggest a possible relationship between polymerization shrinkage and porosity percentage.

The use of tips in the insertion of resin cement into the root canal renders the technique more predictable, producing less formation of bubbles and voids (17). However, void formation might be a result of air entrapment in the material during the mixing process (15). Indeed, for fluid material, indirect void formation is possible through the linking of minor bubbles. Thus, the presence of bubbles does not depend on the type of resin cement used. Moreover, the types of pores identified in the resin cements are classified as mesoporous, having diameters smaller than 20 nanometers (18). The non-significance of the porosity difference among the intra-group root thirds can be explained by the insertion of resin cement using a Centrix syringe in the root canal, allowing for better homogeneity in the distribution of the material. A similar study found that the air bubbles do not derive from a fault in the luting procedure but from the mixing phase of the substrate paste with the catalyst. Indeed, the control of variables, such as the diameter of the root canal and root preparation, promoted proximity to the cementation line (17). This outcome can be explained by the difference in the degree of post-gel contraction and by thin and uniform cement layers in the cementing line producing lower bubble rates (18).

The push-out test used in the present investigation to measure bond strength is a more appropriate methodology to evaluate fiber post adhesion in root dentin (2,5). The main aspect of the push-out test that differentiates it from tensile and shear tests in flat bonded interfaces is the confinement of the composite within the walls of the prepared cavity, which is more akin to the conditions found in the clinic (19). Furthermore, the possibilities of premature failure are reduced, compared to the trimming and non-trimming microtensile tests. To perform the test, some parameters must be observed, such as the size and diameter of the orifice base, in addition to the thickness of the specimens (19).

Dentin matrix is composed mainly of type I collagen and non-collagenous proteins, and these fibrils are intrinsically wet because of their high affinity with water.

The adhesive components are more prone to degradation over time because of water sorption and resin leaching. Initially, ethanol-wet bonding was performed with a progressive ethanol substitution technique, used to chemically dehydrate the demineralized collagen matrix (8). The shrinkage of the collagen fibrils in their lateral dimensions was likely the result of the development of intrafibrillar hydrogen bonding within individual collagen fibrils since ethanol has a lower capacity to break these hydrogen bonds than water (8). Increases in interfibrillar volume are caused by the disruption of shape-maintaining interfibrillar proteoglycan bridges (20). However, the extensive clinical application resulted in the development of a simplified technique (2). The principles of ethanol-wet bonding might be explained using Hoy's triple solubility parameter theory. Hoy's triple solubility parameters consist of dispersive forces (δ_d), polar forces (δ_p), hydrogen bonding forces (δ_h), and total cohesive forces (δ_t) (10). To re-expand matrices, solvents require a higher hydrogen bonding force than $14.8 \text{ (J/cm}^3\text{)}$. Water-wet bonding is capable of re-expanding matrix since water has a very high δ_h value of $40 \text{ (J/cm}^3\text{)}$. However, not all monomers are soluble in water (8). Therefore, ethanol could be an alternative to water because the value of δ_h is $20,0 \text{ J/cm}^3$, resulting in limited collapse of matrices and high resin infiltration into the hybrid layer (10).

Another question concerns the presence of proteoglycan gel in interfibrillar spaces in dentin matrix. EtOH can cause these hydrogels to collapse, and ethanol should be in intimate contact with collagen. This contact should bring the monomers into direct contact with the matrix (20). The results of this study identified a high value for bond strength to ethanol-wet bonding with RelyX ARCTM for the coronal third. These results can be explained by the ability of EtOH to produce interpeptide hydrogen bonding and to promote the maintenance of the interfibrillar spaces, which serve as diffusion channels for resin infiltration (8). The study by Carvalho *et al.* (2009) in ethanol-saturated dentin detected that the diameters of collagen fibrils were smaller than those in water-saturated dentin matrices, leaving larger interfibrillar spaces available for resin impregnation. Because higher bond strength and wider interfibrillar spaces are correlated, the use of ethanol can increase bond strength.

Challenges are imposed when bonding fiber posts to the root canal, especially at the apical area, with adhesive system application and polymerization light access (16), such as a smaller number of dentinal tubules and a greater likelihood of presenting endodontic treatment remnants (4). Another consideration is the configuration factor (C-factor), which is the ratio of the bonded to the unbonded surface areas of cavities. This

factor is very high in root dentin due to the limited amount of unbonded surface area, compared to the area of the bonded surface; hence, the chances for debonding are very high (2). There is also the possibility that the deeper portions are inaccessible to light, and a lower degree of conversion in the apical portion can affect the mechanical properties of resin cement (12). Despite the difficulties found for cementation to the cervical from apical thirds, the study did not find significant differences among root thirds. Corroborating the results of Ekabaram *et al.* (2014), the authors observed after analysis of the data that the bond strength was not affected by the root third factor, different from the results found by Bitter *et al.* (2014), who observed a significant reduction in bond strength in the middle and apical portions of the root canal.

Voids in continuity from the coronal level to the apical level of the interface can cause easier dissolution of the resin cement and dislodgement of post-core restorations under functional stresses. Another negative effect of void formations is that they decrease the bonding strength by restricting the available area for cementation, resulting in shortened survival time of restorations (15). However, the present study did not find a correlation between the volumes of voids and the push-out bond strength data, in agreement with the findings of previous investigations (15). A possible explanation is that, in the apical third, the presence of a thicker smear layer and more remnants of endodontic sealer and gutta-percha in the cervical and middle thirds result in more open dentinal tubules for micromechanical bonding (15). These factors can diminish the negative effects of void parameters on bond strength. However, the results of Silva *et al.* (2015) found that bond strength was affected by the volumes of voids, independent of the region of the root. Indeed, the authors identified that the presence of bubbles and voids promoted stress concentrations at the resin cement/dentin interface and reduced the fiber post bond strength.

Due to improvements in the ability of adhesive systems to adhere to dental tissues, there has been a rise in the frequency of cohesive failures during bond strength testing. This failure prevents the determination of the real interfacial bond strength (21). Therefore, it is important to assess the failure modes of specimens. The most common failure modes observed in this study were adhesive failure between the dentin and resin cement and between the post and resin cement, reinforcing that these interfaces are the locations that experience the greatest stress concentrations and are the most sensible areas for fiber post cementation. This result corroborates another report indicating that the interface was expected to have lower bond strength (18). However, ethanol-wet bonding

with RelyX ARC™ showed the most cohesive failures. The high bond strength for this group might have been responsible for this type of failure (7).

Composite restoratives have the inherent property of polymerization shrinkage, which produces a volumetric change and distributes contractile stresses (14). This shrinkage can be divided into two phases relative to the development of the elastic modulus. In the pre-gel phase, the composites are able to flow, relieving stress within the restoration. After gelation, the composite develops shrinkage stresses because of the advancement of the elastic modulus. Internal shrinkage stresses occur because flow is not able to compensate for the contraction (14). Therefore, the possibility of shrinkage stress and post-gel shrinkage were related, as confirmed by Soares *et al.* (2013), who found a strong correlation between the two factors. The study found a significant difference between the two resin cements, corroborating Braga *et al.* (2002), who explained these differences depending on the characteristics of the polymer network and the overall composition of the resin cement. Post-gel contraction is characterized by the development of internal forces in the material, resulting in molecular rearrangement (viscoelastic properties) and the inability of the material to compensate for the polymerization shrinkage by plastic flow and deformation (24). The magnitude of volumetric shrinkage experienced by a composite is determined by its filler volume fraction and by the composition and degree of conversion of the resin matrix (23). Higher post-gel shrinkage was obtained with the RelyX™ ARC resin cement (0.97%), compared with the RelyX™ U200 cement (0.77%). This result might be explained by the difference in composition of materials, resulting in variations in volumetric shrinkage. According to the manufacturer's information, RelyX™ ARC resin cement has Bis-GMA and TEGDMA as monomers, and RelyX™ U200 has more TEGDMA. It is observed that Bis-GMA has a greater number of covalent bonds and thus better conversion of monomer and greater post-gel polymerization.⁵⁶ According to de Souza *et al.* (2015), acidic monomers have been shown to negatively affect the cement degree of conversion. For this reason, differences in volume fraction, type, molecular weight and functionality might explain the results of the study. However, a lower degree of conversion influences selected mechanical properties, such as bond strength. The degree of conversion was inversely proportional to the concentration of base monomers. The exception was pure TEGDMA, which exhibited low conversion (42%), and Bis-GMA containing polymers, with its conversion ranging from 54% to 85% (25). However, this study indicated that increasing the diluent monomer concentration decreases the leachable fraction. Other factors that

could be influenced by the bond strength are elastic modulus and Vickers hardness. The Vickers hardness and degree of conversion of resin cements are directly related, while the elastic modulus is directly related to the Vickers hardness and polymerization stress (1). These factors might explain why RelyX™ U200 had the lowest values of bond strength.

CONCLUSION

Within the limitations of this study, the following conclusions were drawn.

- 1- The resin cement porosity was lower when pretreatment was performed with ethanol-wet bonding with RelyX™ U200.
- 2- The bond strength was influenced by the pretreatment and the resin cement used for fiber post cementation. The highest bond strength was obtained when ethanol-wet bonding was associated with RelyX™ ARC.
- 3- RelyX™ ARC exhibited higher post-gel shrinkage than RelyX™ U200 resin cement.

RESUMO

Este estudo *in vitro* objetivou avaliar os efeitos de diferentes pré-tratamentos para adaptação de pinos de fibra de vidro cimentados em canais radiculares com diferentes cimentos resinosos sobre a porcentagem de porosidade e resistência de união. Doze raízes de incisivo bovino (n=12) foram preparados com broca largo. Após preparo do conduto, os espécimes foram divididos aleatoriamente de acordo com o tratamento de superfície radicular: técnica convencional e técnica alcoólica simplificada. Dois tipos de cimentos resinosos foram usados para cimentação dos pinos: cimento resinoso dual com sistema adesivo de 3 passos (RelyX™ ARC / Scotchbond Multipurpose Plus) e um cimento resinoso dual autoadesivo (RelyX™ U200). Após a cimentação dos pinos, as raízes foram armazenadas em 100% de umidade à temperatura ambiente por sete dias. As amostras foram digitalizadas através de microtomografia computadorizada (micro-CT). O software NRecon foi utilizado para gerar imagens tridimensionais, e o software CTan foi utilizado para analisar a porcentagem de porosidade (%) na interface de cimentação. Para avaliação da resistência de união, as raízes foram cortadas em série e as fatias foram submetidas a um teste push-out. Adicionalmente, a contração pós-gel dos cimentos resinosos (%) foram mensuradas pelo método strain-gauge (n = 10). As raízes preparadas para técnica alcoólica simplificada com RelyX™ U200 apresentaram porosidade significativamente menor para os terços cervical e apical (p <0,05). O grupo preparado

para técnica alcoólica simplificada com RelyX™ ARC apresentou melhores resultados de resistência de união para os terços cervical e apical ($p < 0,05$). O RelyX™ ARC (0,97%) produziu um maior valor de encolhimento pós-gel do que o RelyX™ U200 (0,77%). Quando os canais foram pré-tratados com a técnica alcoólica simplificada, houve melhores resultados na porcentagem de porosidade e força de adesão.

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Table 1 – Resin cements, manufacturers, composition and application mode.

Resin Cement	Composition	Manufacturer	Application instruction
RelyX ARC/ Scotchbond Multipurpose Plus	Silicon-treated ceramic, triethylene glycol dimethacrylate (TEGDMA), bisphenol A diglycidyl methacrylate (bis-GMA); silicon-treated silica, functionalized dimethacrylate polymer Primer: HEMA, polyalkenoic acid copolymer, water Adhesive: Bis-GMA, HEMA, tertiary amines, photo-initiator	3M ESPE	Mix the two pastes in a ration of 1:1 for 10 seconds, mixture applied on root canal and, after the post were slowly seated, light-activated for 40 seconds in three different inclinations. Remove excess of cement before the light-activated • Apply 1 coat of primer, • Blow gently for 5 s • Apply 1 coat of adhesive • Light cure for 10 seconds
RelyX U200 (U200)	Base paste: Methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers, rheological additives Catalyst paste: Methacrylate monomers, alkaline fillers, silanated fillers, initiator components, stabilizers, pigments, rheological additives	3M ESPE	Mix the two pastes in a ration of 1:1 for 10 seconds, mixture applied on root canal and, after the post were slowly seated, light-activated for 40 seconds in three different inclinations. Remove excess of cement before the light-activated

Table 2. Means $\pm$ SD of total porosity in interface measured in coronal, middle, and apical thirds for pre-treatment and resin cements

Location	Pre-treatment			
	RelyX ARC		RelyX U200	
	WBD	EBD	WBS	EBS
Coronal	0.71 (0.01) ^{Aa}	0.54 (0.02) ^{Ab}	0.62 (0.02) ^{Ac}	0.50 (0.01) ^{Ad}
Middle	0.70 (0.02) ^{Aa}	0.54 (0.03) ^{Ab}	0.63 (0.03) ^{Ac}	0.50 (0.02) ^{Ab}
Apical	0.72 (0.03) ^{Aa}	0.56 (0.02) ^{Ab}	0.59 (0.04) ^{Ab}	0.50 (0.03) ^{Ac}

Means followed by equal superscript uppercase letters did not differ in comparisons by rows.

Means followed by equal superscript lowercase letters did not differ in comparisons by columns.

Table 3. Means $\pm$ standard deviations bond strength values (MPa) measured in coronal, middle, and apical thirds for pre-treatment and resin cements

Location	Pre-treatment			
	RelyX ARC		RelyX U200	
	WBD	EBD	WBS	EBS
Coronal	11.5 (5.3) ^{Aa}	20.1 (2.4) ^{Ab}	5.1 (3.4) ^{Ac}	4.3 (1.6) ^{Ac}
Middle	7.3 (4.8) ^{Aa}	10.5 (4.8) ^{Aa}	6.2 (3.4) ^{Aa}	8.2 (2.3) ^{Aa}
Apical	7.8 (5.5) ^{Aa}	12.6 (3.8) ^{Aa,b}	4.1 (2.0) ^{Aa,c}	8.0 (2.5) ^{Aa}

Means followed by equal superscript uppercase letters did not differ in comparisons by rows.

Means followed by equal superscript lowercase letters did not differ in comparisons by columns.

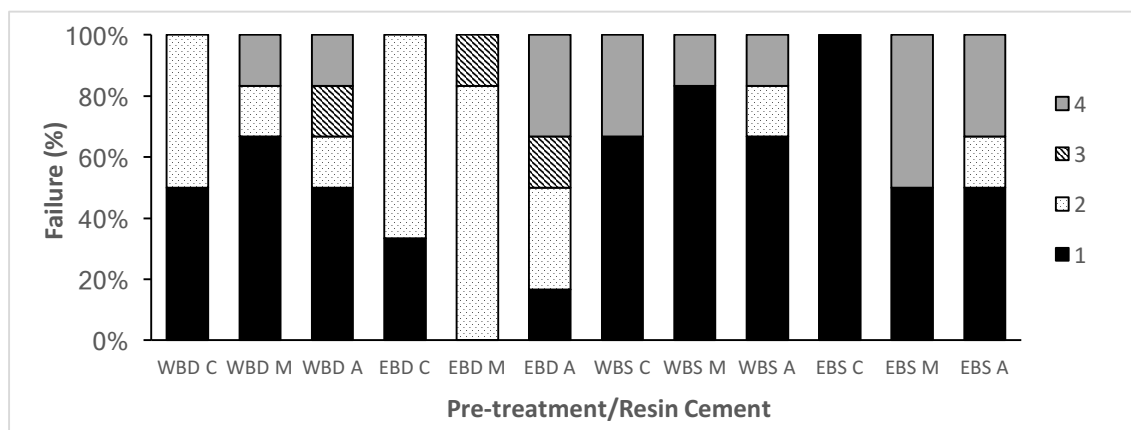
Table 4. Means $\pm$ SD for Post-gel shrinkage of the two resin cements

Resin Cements	RelyX TM ARC	RelyX TM U200	p-value*
Post-gel shrinkage	0.97 $\pm$ 0.15%	0.77 $\pm$ 0.026%	0.001

Legends

Figure 1. Failure modes found in different pre-treatment/ resin cement for different root levels. 1= adhesive failure 2=cohesive failure in cement 3=cohesive failure in dentin 4=mixed failure. Bottom legends refer to pre-treatment and resin cement for root levels. WBD C, Water wet-bonding with RelyX™ ARC in coronal third; WBD M, Water wet-bonding with RelyX™ ARC in middle third; WBD A, Water wet-bonding with RelyX™ ARC in apical third; EBD C: Ethanol wet-bonding with RelyX™ ARC in coronal third; EBD M: Ethanol wet-bonding with RelyX™ ARC in middle third; EBD A: Ethanol wet-bonding with RelyX™ ARC in apical third; WBS C, Water wet-bonding with RelyX™ U200 in coronal third; WBS M, Water wet-bonding with RelyX™ U200 in middle third; WBS A, Water wet-bonding with RelyX™ U200 in coronal apical; EBS C: Ethanol wet-bonding with RelyX™ U200 in coronal third; EBS M: Ethanol wet-bonding with RelyX™ U200 in middle third; EBS A: Ethanol wet-bonding with RelyX™ U200 in apical third.

Figure 1.



ARTIGO 2

*Title page

Effect of irrigation protocol on bond strength and durability of the adhesion of fiber posts to root dentin

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* Artigo formatado de acordo com as normas do periódico *Brazilian Oral Research* (ANEXO)

ABSTRACT

This study analyze the effects of ethanol pretreatment on the bond strength over time of two resin cements to intraradicular dentin. Forty-four bovine teeth were endodontically treated. After post-space preparation, the specimens were randomized into two types of pretreatment groups: water-wet bonding and ethanol-wet bonding. Two types of resin cement were used to lute the fiber posts: dual conventional resin cement with a 3-step etch-and-rinse adhesive system and self-adhesive resin cement. The bond strength was evaluated at each root third (coronal, middle and apical) using a push-out test both 24 h after cementation and after 12 months of storage in distilled water. Data were analyzed by Kruskal-Wallis and Mann-Whitney tests ($p \leq 0.05$). Bond strength was significantly affected by the pretreatment ($p < 0.05$), storage time ($p < 0.05$) and tested root third ($p < 0.05$). Higher bond strength values were observed after 24 h without significant differences among treatment groups or among the root thirds. After 12 months, the middle third root in the water-wet bonding with conventional etch-and-rinse resin cement group revealed the lowest bond strength compared to the other groups. After 12 months, the simplified ethanol-wet bonding with self-adhesive resin cement bond strengths were higher than those of the water-wet bonding with self-adhesive resin cement groups in the middle and apical thirds. Pretreatment with 100% ethanol for 60 s increased the bond strength of self-adhesive resin cement but did not influence conventional resin cement performance. Storage for 12 months produced a negative effect for all treatment groups and root thirds.

Keywords: bond strength; intraradicular dentin; fiber post; ethanol-wet bonding

INTRODUCTION

The use of adhesively luted fiber posts has become popular¹ and is routinely performed in daily practice² to restore endodontically treated teeth.³ The esthetics and the modulus of elasticity of fiber posts are similar to those of dentin, which represent some of the advantages of fiber posts.⁴ Because the modulus of elasticity is similar to that of dentin, fiber posts provide a homogeneous dissipation of loading stresses to the tooth structure⁵ and reduce the risk of root fractures.⁶ However, according to a systematic review, loss of retention is a major mode of failure for fiber posts bonded with resin cements.⁷ Some of the mechanisms that could be responsible for bond failure at the resin-dentin interfaces include the hydrolytic degradation of the cured resin by water sorption⁸ and the enzymatic degradation of vulnerable collagen fibers,⁹ due to the activation of endogenous dentin matrix metalloproteinases (MMPs).¹⁰ The organic components of dentin hinder its adhesion with resin composites.¹¹ Some studies have evaluated the effects of different bonding approaches and luting systems on the bond strength between fiber posts^{2,12,13} and root dentin.¹⁴

Different protocols can be used with dual-cured resin cements, such as etch-and-rinse, self-etch and universal adhesive systems.³ The acid etching used in etch-and-rinse systems (total etching systems) demineralizes the dentin surface to expose a type I collagen fibril network, providing a space for resin infiltration.¹⁵ In brief, self-etching systems are composed of acidic monomers that modify and integrate with the hybrid layer, simultaneously.¹⁶ Self-adhesive resin cements are composed of acid groups that are able to chemically bond with calcium ions present in the tooth.¹⁷ However, adhesive monomers are not able to fully encapsulate the exposed collagen matrix, leaving partially exposed collagen fibrils in the hybrid layer that lack the protection of the polymerized

resin.⁹ The presence of water leaves demineralized collagen fibrils vulnerable and contributes to the reduction of bond strength over time.¹⁸

Therefore, protocols to counter the mechanisms of degradation have been developed to improve the durability of bonding strength.^{10,19} Ethanol-wet bonding has been proposed by Pashley *et al.*, 2007.²⁰ The goal of this protocol is to dilute and displace all of the water present in the acid-etched dentin with ethanol, leaving the unsupported collagen fibrils in an ethanol-moist, rather than water-filled, environment to allow monomer resins to impregnate the substrate.²⁰ For clinical purposes, the simplified ethanol-wet bonding protocol (without stepwise ethanol applications) has shown promising results.^{12,21} However, few studies have been conducted using this protocol to lute fiber posts to the root canal with self-adhesive resin cement.^{2,22}

The aim of the present study was to examine the effects of simplified ethanol-wet bonding on the bond strength of fiber posts to intraradicular dentin, over time, using conventional and self-adhesive resin cements. The null hypotheses tested were that the bond strength of fiber posts to dentin would not be affected by either (i) protocol (ii) storage, or (iii) root third.

MATERIALS AND METHODS

A total of 44 freshly extracted bovine incisor teeth, with homogeneous shapes and sizes, were collected, according to the guidelines of the Animal Ethics Committee of the Institution (protocol #31/2017). After extraction, the teeth were stored in a 0.1% thymol solution for up to three months. The solution was changed every week. The crowns were removed using a diamond observed on a precision cutting machine (Isomet 1000; Buehler), under constant water irrigation. The obtained roots measured 15 mm.

A single, trained operator prepared the root canals. The canals were endodontically prepared using rotary instruments (Xmart; Dentsply Maillefer), under irrigation with 2.5% sodium hypochlorite (Asfer Indústria Química Ltda). Final irrigation was performed with trisodium ethylenediamine tetra acetic acid (EDTA; Biodinâmica) for 3 minutes, followed by rinsing with water and drying with absorbent paper points. The roots were obturated by a vertical condensation technique, using gutta-percha cones and AH Plus cement (Dentsply Maillefer) and were then stored in 100% humidity at 37°C for 7 days.^{23,24}

The root canals were prepared using Largo burs #3 and #4 (Maillefer- Dentsply, Petrópolis, RJ, Brazil), with a slow rotary speed. The instrumentation length was 11 mm, and 4 mm of filled apical canal was left. The glass fiber post used was a Reforpost RX #2 (Angelus), and two cementation systems were evaluated (Table 1): an etch-and-rinse 3-step adhesive system (Scotchbond Multipurpose Plus; 3M ESPE), with a conventional dual polymerizing resin cement (RelyX ARC; 3M ESPE), and a self-adhesive resin cement (RelyX U200; 3M ESPE). The prepared roots were randomly divided into four groups (n=11), according to the protocol used: water-wet bonding with conventional etch-and-rinse resin cement (WBD), simplified ethanol-wet bonding with conventional etch-and-rinse resin cement (EBD), water-wet bonding with self-adhesive resin cement (WBS) and simplified ethanol-wet bonding with self-adhesive resin cement (EBS).

Before cementation, the glass fiber posts were prepared. Posts were immersed in 35% hydrogen peroxide (FGM Produtos Odontológicos) for one minute, rinsed with water for one minute, and dried with compressed air. This process was followed by the application of a silane-coupling agent for one minute (Silano; Angelus).²⁵ All resin cements were light-activated for 40 s in the buccal, lingual and incisal angulations, using a quartz-tungsten halogen light (600 mW/cm², Demetron 501; Kerr Corporation).²⁴ The

roots were covered with black tape at the time of cementation to prevent the influence of ambient light and to simulate the oral environment.⁴

For the water-wet bonding groups, the root canals were rinsed with distilled water after etching with either 37% phosphoric acid (FGM Produtos Odontológicos) for 15 s (WBD) or 2.5% sodium hypochlorite (WBS) for 60 s, and the excess was removed from the post space using absorbent paper points. In the ethanol-wet bonding groups, the root canal was rinsed with distilled water after etching with either 37% phosphoric acid for 15 s (EBD) or 2.5% sodium hypochlorite (EBS) for 60 s, the excess water was removed with absorbent papers, the root canals were completely filled with 100% ethanol for 60 s, and the excess ethanol was gently removed.²²

After root dentin treatment, the glass fiber posts were luted with the resin cements. The bonding agent and the resin cements were used according to the manufacturer's instructions (Table 1). After cementation procedures, roots were stored in a 100% humidity environment at 37°C for 24 h or 12 months.

Bovine roots (n=11) were fixed in 20 mm X 20 mm acrylic plates with sticky wax and sectioned perpendicular to the long axis of the fiber post to produce two slices, approximately 1 mm thick, for each root region (apical, middle and coronal), using a precision cutting machine under water cooling (Isomet 1.000 Precision Saw). Each slab was marked on its apical side with an indelible marker, and the thickness was measured using a digital caliper (Mitutoyo Digimatic Caliper Serie 500, Mitutoyo Sul Americana). Seven slices were obtained from each root canal. The first slice from each specimen was excluded.

The specimens were subjected to the push-out bond test, using a 1 mm diameter plunger, and the load was applied at a cross-head speed of 0.5 mm/min in an apical-coronal direction using a universal test machine (EZ-LX Long-Stroke Model). The

plunger was centralized to avoid contact with the dentin. The bond strength was calculated in megapascals (MPa) by dividing the load, recorded in Newtons (N), by the area (mm²) of the post/dentin interface.^{22,24}

The failure mode was observed using a stereomicroscope at 40X magnification. The failure mode was assessed by two calibrated operators, and each sample was categorized as follows: adhesive failure (failure at the cement-dentin or cement-post interface), mixed, or cohesive failure (failure within the cement).^{22,24}

To analyze the cementation line using scanning electron microscopy (SEM), 2 representative specimens from each experimental group (24 h and 12 months) were evaluated. The surfaces were polished, demineralized, deproteinized, dehydrated, and coated with a carbon layer under medium-vacuum.⁴ The images were obtained with low-vacuum scanning electron microscopy (Quanta 200F; FEI Co) at 70X, 600X, and 1000X magnification.

For statistical analyses, push-out bond strength data were analyzed using a statistical package (IBM SPSS Statistics 21). The alpha (Type I) error was set at 0.05. A Kruskal-Wallis and a post hoc test (Mann-Whitney), with push-out bond strength as the dependent variable and protocol, storage time and root third as the independent factors, were applied at four (WBD, WBS, EBD, and EBS), two (24 h and 12 months) and three levels (coronal, middle, and apical). Failure modes were evaluated using crosstabs and the chi-squared test.

RESULTS

The median and interquartile distance of the push-out bond strengths obtained from the experimental groups are shown in Table 2. The median push-out bond strength (MPa) was significantly affected by the protocol used ($p < 0.05$), the storage time ($p < 0.05$)

and the tested root third ($p < 0.05$). Higher bond strength values were observed after 24 h, regardless of the bonding protocol used or the root third tested.

The 12-month storage time produced a negative effect on bonding protocols and root thirds. The bonding strengths of the water-wet bonding with conventional etch-and-rinse resin cement (WBD) groups were similar to those of the ethanol-wet bonding with self-adhesive resin cement groups for all root sections. The middle third root section of the water-wet bonding with self-adhesive resin cement group revealed the lowest bond strength (2.2 ± 1.4 MPa) when compared to the other groups. The bonding strength of ethanol-wet bonding with self-adhesive resin cement was greater than that of water-wet bonding with self-adhesive resin cement for the middle and apical thirds. The cervical third presented higher bonding strength values for all groups, except for the ethanol-wet bonding with self-adhesive resin cement group.

The distribution of the failure modes of the debonded dentin specimens from each group after 24 h or 12 months of storage are shown in Figure 1. The specimens from both the water and ethanol-wet bonding groups demonstrated a predominant adhesive failure pattern, followed by a mixed failure pattern for both 24 h and 12 months.

Representative SEM images showed a homogeneous hybrid layer and evident resin tags in samples 24 h after conventional etch-and-rinse resin cement treatment, irrespective of the wet-bonding protocol used, in the coronal and middle thirds (Fig. 2). Irregular, juxtaposed cement lines, without an evident hybrid layer or resin tags was observed after conventional etch-and-rinse resin cement in the apical third after 24 h, in all samples that received self-adhesive resin cement treatment, and for all samples that were stored for 12 months (Fig. 3).

DISCUSSION

A clinical, hypothetical model was used to investigate the effect of bonding protocols, using different dual cured resin cements, on bond strength with bovine root dentin.

There were no observed effects for the bonding protocol used or the root third examined on bond strength after 24 h of storage. The low moisture level in the root canal²⁶ could be responsible for the diminished effect of water hydrolysis,^{18,27} and a 24-hour storage time may not be sufficient to degrade the adhesive interface, especially the hybrid layer.²⁸ The results of the present study are in accordance with existing literature, which reported that, regardless of the cementation protocol used, optimal results for bonding effectiveness are found after 24 h, and there are no beneficial effects of using an ethanol-wet bonding protocol.^{2,29,30} Although there were no differences in the bond strength results, SEM analysis showed different patterns of hybrid layer formation. For dual cured resin cement with an etch-and-rinse approach, a uniform hybrid layer and long resin tags were observed.¹² For self-etch resin cement, a thin and uninformed hybrid layer was observed.^{12,31} It has been observed that differences in the hybrid layer do not directly affect the bonding strength. However, it has been reported that the application of ethanol to the root canal, after the application of an adhesive system and dual cured resin cement, could improve the bonding strength of the resin to root dentin.^{1,32}

Controversy exists concerning the bond strength values in different root thirds after 24 h of storage. Several studies have reported a decreased bond strength in the coronal third compared to the apical third,³³ and other studies did not show any effect for root thirds.³⁴ Root thirds did not affect the bond strength results in our study. It has been hypothesized that light transmitted through the glass fiber post is able to adequately convert monomers into polymers along the resin cement length and thickness.²⁹

The bond strength results significantly decreased after 12 months of storage. Both the bonding protocol used and the root dentin third examined negatively affected the bond strength results. Therefore, the null hypothesis of the present study can be partially rejected. Hydrophilic resin components and the degradation of collagen fibrils may be able to decrease the dentin bond strength over time.^{9,14,18} The chemical composition of the materials used in our study includes hydrophilic monomers, such as HEMA (etch-and-rinse adhesive system), and acidic monomers (self-etch resin cement). The presence of hydrophilic components could promote the hydrolysis of monomers,^{19,27} leading to their elution from the resin materials. As a result, the integrity of the hybrid layer would be compromised, and collagen fibrils would be exposed, causing the degradation of collagenolytic and gelatinolytic activities.¹⁹ Endogenous matrix metalloproteinases (MMPs) and dentin cysteine cathepsins are enzymes that are responsible for the degradation of exposed collagen fibrils.^{9,27} Our results are in accordance with previous studies, which reported a significant decrease in resin-dentin bonding, even when using an ethanol-wet bonding protocol.¹⁹

Differences in bonding mechanisms have resulted in differences in mechanical properties, when comparing conventional etch-and-rinse and self-adhesive resin cements.³¹ RelyX U200 is classified as a “mild” self-etch resin, with a pH of 2.³⁵ The composition of self-adhesive resin cements usually presents functional monomers, such as methacrylate phosphoric esters with carboxylic or phosphoric acid groups, that react with the hydroxyapatite of the tooth tissue.¹⁷ These monomers could demineralize dentin without forming a distinct hybrid layer.³ In the present study, the self-adhesive resin cement, applied according to the conventional bonding protocol, demonstrated the lowest bond strength value after 12 months, which is in accordance with a previous study.³⁶ The low bond strength values could be attributed to the scarce penetration of the resin cement

into dentinal tubules and to the poor formation of a hybrid layer and resin tags (Fig. 3). The functional monomers might not be able to dissolve the smear layer created on root canal walls during the post space preparation.² In addition, self-adhesive resin cements have demonstrated a low degree of conversion, likely due to the presence of acidic monomers in their composition,³⁷ and an increased viscosity during the polymerization reaction.¹⁷

After 12 months of storage, for the self-adhesive resin cement, the use of the ethanol-wet bonding protocol improved the bonding performance when compared to the results of the water-wet bonding protocol. It has been speculated that when ethanol is used to replace water in collagen fibrils, the spaces between fibrils become filled with ethanol, which leads to reduced moisture in the substrate, allowing for the enhanced monomer infiltration into the dentin matrix.¹¹ When water-wet bonding is used, the residual water might cause phase separation^{20,38} and reduced polymerization rates for the adhesive.¹⁰

Analyses of the failure modes in the present investigation also demonstrated an increased rate of adhesive failure between 24 h and 12 months for all experimental groups. This increase could be related to the reduction in bond strength and the potential degradation of the resin-dentin interface.¹³ Indeed, the bonded interface could be affected by residual oxygen.⁶ Moreover, bond strengths were significantly reduced in the middle and apical thirds for most of the experimental groups, except for EBS. Tubular density and the diameter of tubules are important for debonding.³⁹ It has been observed that apical dentin is less organized and more irregular, leading to a diminished hybrid layer.³ These differences in the dentin structure could play a role in these relationships.⁴⁰ However, the EBS groups that were pretreated with ethanol also showed no significant differences among root thirds. Ethanol application could contribute to moisture control in the depth

of the root canal and to a deeper penetration of adhesive monomers.³⁸ Furthermore, it is assumed that evaporation of the solvent is more difficult for complex preparations, such as in the post space.²

CONCLUSIONS

The following conclusions were drawn:

1. Pretreatment with 100% ethanol for 60 s increased the bond strength of self-adhesive resin cement but did not influence conventional resin cement performance.
2. Storage for 12 months produced a negative effect for all groups and root thirds.
3. Except for self-adhesive resin cement with ethanol-wet bonding, the apical regions presented the lowest bond strength results.

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Table 1. Dual cure resin cements used

Resin Cement	Composition	Manufacturer	Application instruction
RelyX ARC/ Adper Scotchbond Multipurpose Plus	Paste A: Silicon-treated ceramic, triethylene glycol dimethacrylate (TEGDMA), bisphenol A diglycidyl methacrylate (Bis-GMA); silane-treated silica, functionalized dimethacrylate polymer, 2-benzotriazolyl-4-methylphenol, 4-(dimethylamino)-benzene ethanol Paste B: silane-treated ceramic, TEGDMA, bis-GMA, silane-treated silica, functionalized dimethacrylate polymer, 2-benzotriazolyl-4-methylphenol, benzoyl peroxide Primer: HEMA, polyalkenoic acid copolymer, water Adhesive: Bis-GMA, HEMA, tertiary amines, photo-initiator	3M ESPE	Mix the two pastes in a ration of 1:1 for 10 seconds, mixture applied on root canal and, after the post were slowly seated, light-activated for 40 seconds in three different inclinations. Remove excess of cement before the light-activated • Apply 1 coat of primer, • Blow gently for 5 s • Apply 1 coat of adhesive • Light cure for 10 seconds
RelyX U200	Base paste: Methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers, rheological additives Catalyst paste: Methacrylate monomers, alkaline fillers, silanated fillers, initiator components, stabilizers, pigments, rheological additives	3M ESPE	Mix the two pastes in a ration of 1:1 for 10 seconds, mixture applied on root canal and, after the post were slowly seated, light-activated for 40 seconds in three different inclinations. Remove excess of cement before the light-activated

Table 2: Push-out bond strength of fiber posts bonded to intraradicular dentine after 24h and 12 months of ageing

Group	Immediate			12 months		
	Coronal	Middle	Apical	Coronal	Middle	Apical
WBD	10.9(5.6) ^{A,a}	7.8(7.3) ^{A,a}	10.6(6.4) ^{A,a}	7.5(4.2) ^{B,b}	3.4(2.5) ^{C,bcd}	3.2(3.0) ^{C,bc}
EBD	12.5(5.2) ^{A,a}	9.4(6.4) ^{A,a}	13.5(9.2) ^{A,a}	4.6(2.1) ^{B,b}	3.3(1.8) ^{C,c}	3.0(2.2) ^{C,c}
WBS	9.0(7.0) ^{A,a}	9.8(5.6) ^{A,a}	9.4(3.5) ^{A,a}	2.9(1.6) ^{B,c}	2.2(1.4) ^{C,d}	2.3(1.0) ^{C,c}
EBS	11.2(3.8) ^{A,a}	10.5(4.9) ^{A,a}	11.4(7.8) ^{A,a}	6.9(3.6) ^{B,b}	7.0(4.7) ^{B,b}	6.0(4.9) ^{B,b}

Median and interquartile distance in MPa. Median followed by different letters (uppercase in the row and lowercase in the columns) are significant different ($P < 0.05$).

Figure 1: Distribution of failure modes after 24 h and 12 months

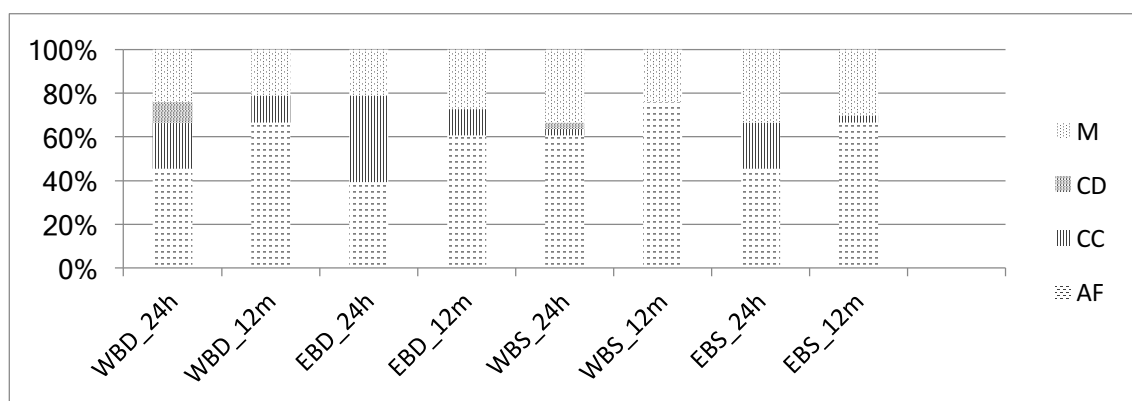


Figure 2. Representative scanning electron micrographs for conventional etch-and-rinse resin cement after 24 hours of storage. A, (WBD) coronal third. B, (WBD) middle third. C, (EBD) coronal third. D, (EBD) middle third. P, post; C, resin cement; H, hybrid layer.

*Resin tags.

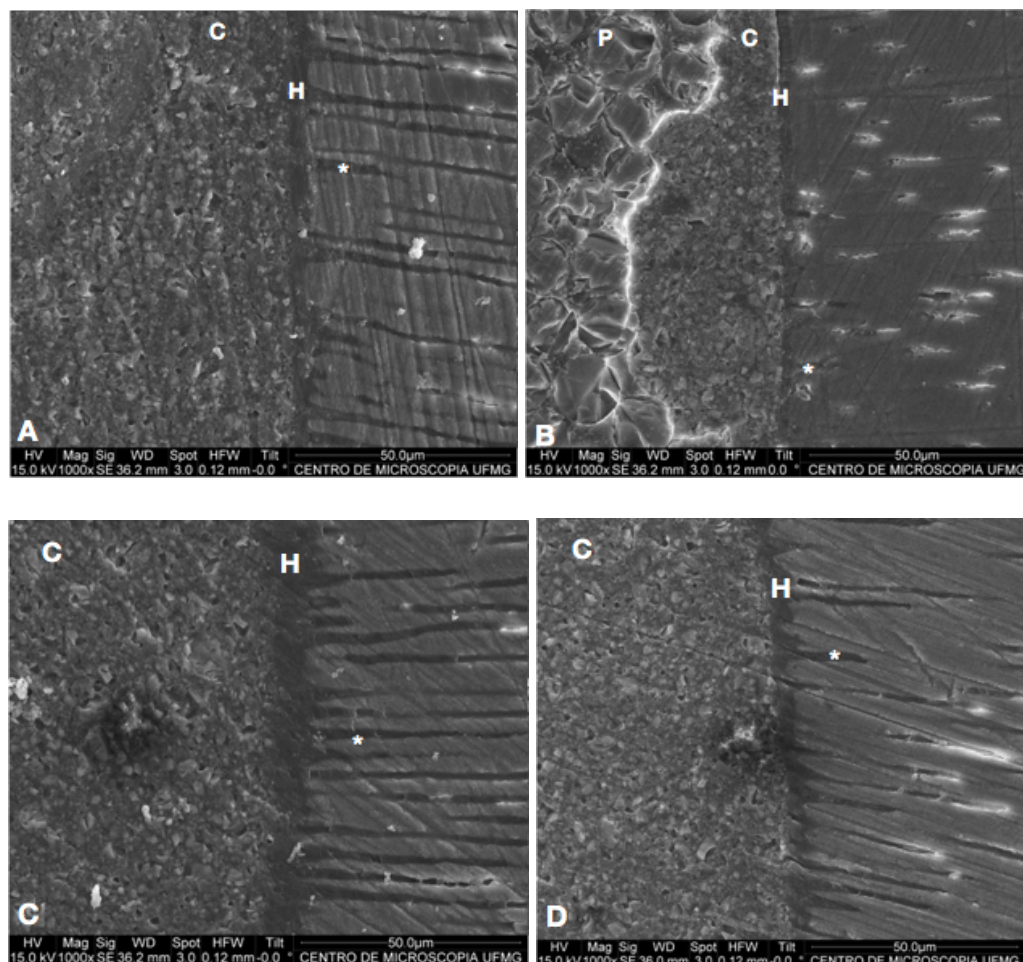
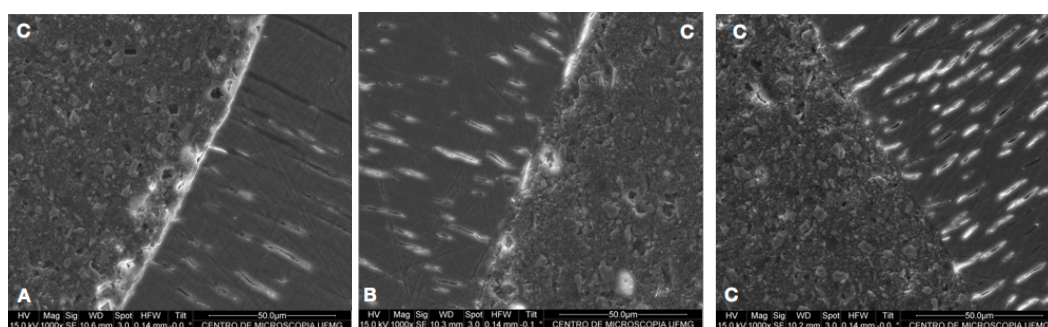


Figure 3. Representative scanning electron micrographs for self-adhesive resin cement for EBS after 12 months of storage. A, coronal third. B, middle third. C, apical third.



4 CONSIDERAÇÕES FINAIS

Dentro das limitações do estudo, as conclusões encontradas foram:

- 1- O cimento resinoso RelyX™ U200 com a técnica alcoólica simplificada apresentou a menor porcentagem de porosidade nos terços cervical e apical.
- 2- A resistência de união imediata não foi influenciada pelo fator terço radicular e tratamento de superfície interna/cimento resinoso. Diferente da resistência de união após 12 meses, que apesar do efeito negativo para todos os grupos e terços radiculares, o uso da técnica alcoólica simplificada com o cimento resinoso autoadesivo produziu melhores resultados.
- 3- A área de união após 24 horas, apresentou evidentes *tags* de resina e homogeneidade da camada híbrida para RelyX™ ARC independente do tipo de pré-tratamento, não sendo observado para RelyX™ U200. Após 12 meses, irregularidades e deficiência na manutenção da camada híbrida foi observado para os diferentes cimentos e pré-tratamento.
- 4- O cimento resinoso RelyX™ U200 apresentou o menor grau de contração de polimerização pós-gel.

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ANEXO - Normas para publicação

NORMAS PARA PUBLICAÇÃO NO PERIÓDICO BRAZILIAN DENTAL JOURNAL

Escopo e política

O Brazilian Dental Journal é um periódico científico revisado por pares (sistema duplo-cego) que publica Documentos Originais Completos, Comunicações Curtas, Relatórios de Casos e Críticas Convidadas, tratando os diversos campos da Odontologia ou áreas relacionadas, com acesso aberto. Serão considerados para publicação apenas artigos originais. Na submissão de um manuscrito, os autores devem informar em carta de encaminhamento que o material não foi publicado anteriormente e não está sendo considerado para publicação em outro periódico, quer seja no formato impresso ou eletrônico.

ENDEREÇO ELETRÔNICO PARA SUBMISSÃO:

<http://mc04.manuscriptcentral.com/bdj-scielo>

SERÃO CONSIDERADOS APENAS TRABALHOS REDIGIDOS EM INGLÊS. Autores cuja língua nativa não seja o Inglês, devem ter seus manuscritos revisados por profissionais proficientes na Língua Inglesa. Os trabalhos aceitos para publicação serão submetidos à Revisão Técnica, que compreende revisão lingüística, revisão das normas técnicas e adequação ao padrão de publicação do periódico. O custo da Revisão Técnica será repassado aos autores. A submissão de um manuscrito ao BDJ implica na aceitação prévia desta condição. A decisão de aceitação para publicação é de responsabilidade dos Editores e baseia-se nas recomendações do corpo editorial e/ou revisores "ad hoc". Os manuscritos que não forem considerados aptos para publicação receberão um e-mail justificando a decisão. Os conceitos emitidos nos trabalhos publicados no BDJ são de responsabilidade exclusiva dos autores, não refletindo obrigatoriamente a opinião do corpo editorial.

Todos os manuscritos serão submetidos a revisão por pares. Autores e revisores serão mantidos anônimos durante o processo de revisão. Os artigos aceitos para a publicação se tornam propriedade da revista.

Brazilian Dental Journal é um jornal de acesso aberto, o que significa que todos os artigos publicados estão disponíveis gratuitamente na Internet imediatamente após a publicação.

O Brazilian Dental Journal manterá os direitos autorais e editoriais de todos os artigos publicados, incluindo traduções. Os usuários podem usar, reutilizar e construir sobre o material publicado na revista, mas apenas para fins não comerciais e desde que a fonte seja claramente e adequadamente mencionada.

A Revista adota sistema para identificação de plágio (AntiPlagiarist - ACNP Software).

O Brazilian Dental Journal está indexado na base de dados DOAJ para acesso público.

Forma e preparação de manuscritos

AS NORMAS DESCRITAS A SEGUIR DEVERÃO SER CRITERIOSAMENTE SEGUIDAS.

GERAL

Submeter o manuscrito em Word e em PDF, composto pela página de rosto, texto, tabelas, legendas das figuras e figuras (fotografias, micrografias, desenhos esquemáticos, gráficos e imagens geradas em computador, etc).

O manuscrito deve ser digitado usando fonte Times New Roman 12, espaço entrelinhas de 1,5 e margens de 2,5 cm em todos os lados. NÃO UTILIZAR negrito, marcas d'água ou outros recursos para tornar o texto visualmente atrativo.

As páginas devem ser numeradas seqüencialmente, começando no Summary.

Trabalhos completos devem estar divididos sequencialmente conforme os itens abaixo:

1. Página de Rosto
2. Summary e Key Words
3. Introdução, Material e Métodos, Resultados e Discussão
4. Resumo em Português (obrigatório apenas para os autores nacionais)
5. Agradecimentos (se houver)
6. Referências
7. Tabelas
8. Legendas das figuras
9. Figuras

Todos os títulos dos capítulos (Introdução, Material e Métodos, etc) em letras maiúsculas e sem negrito.

Resultados e Discussão NÃO podem ser apresentados conjuntamente.

Comunicações rápidas e relatos de casos devem ser divididos em itens apropriados.

Produtos, equipamentos e materiais: na primeira citação mencionar o nome do fabricante e o local de fabricação completo (cidade, estado e país). Nas demais citações, incluir apenas o nome do fabricante.

Todas as abreviações devem ter sua descrição por extenso, entre parênteses, na primeira vez em que são mencionadas.

PÁGINA DE ROSTO

A primeira página deve conter: título do trabalho, título resumido (short title) com no máximo 40 caracteres, nome dos autores (máximo 6), Departamento, Faculdade e/ou Universidade/Instituição a que pertencem (incluindo cidade, estado e país). NÃO INCLUIR titulação (DDS, MSc, PhD etc) e/ou cargos dos autores (Professor, Aluno de Pós-Graduação, etc).

Incluir o nome e endereço completo do autor para correspondência (informar e-mail, telefone e fax).

A página de rosto deve ser incluída em arquivo separado do manuscrito.

MANUSCRITO

O manuscrito deve conter:

A primeira página do manuscrito deve conter: título do trabalho, título resumido (short title) com no máximo 40 caracteres, sem o nome dos autores.

SUMMARY

A segunda página deve conter o Summary (resumo em Inglês; máximo 250 palavras), em redação contínua, descrevendo o objetivo, material e métodos, resultados e conclusões. Não dividir em tópicos e não citar referências.

Abaixo do Summary deve ser incluída uma lista de Key Words (5 no máximo), em letras minúsculas, separadas por vírgulas.

INTRODUÇÃO

Breve descrição dos objetivos do estudo, apresentando somente as referências pertinentes. Não deve ser feita uma extensa revisão da literatura existente. As hipóteses do trabalho devem ser claramente apresentadas.

MATERIAL E MÉTODOS

A metodologia, bem como os materiais, técnicas e equipamentos utilizados devem ser apresentados de forma detalhada. Indicar os testes estatísticos utilizados neste capítulo.

RESULTADOS

Apresentar os resultados em uma seqüência lógica no texto, tabelas e figuras, enfatizando as informações importantes.

Os dados das tabelas e figuras não devem ser repetidos no texto.

Tabelas e figuras devem trazer informações distintas ou complementares entre si.

Os dados estatísticos devem ser descritos neste capítulo.

DISCUSSÃO

Resumir os fatos encontrados sem repetir em detalhes os dados fornecidos nos Resultados.

Comparar as observações do trabalho com as de outros estudos relevantes, indicando as implicações dos achados e suas limitações. Citar outros estudos pertinentes.

Apresentar as conclusões no final deste capítulo. Preferencialmente, as conclusões devem ser dispostas de forma corrida, isto é, evitar citá-las em tópicos.

RESUMO (em Português) - Somente para autores nacionais

O resumo em Português deve ser IDÊNTICO ao resumo em Inglês (Summary). OBS: NÃO COLOCAR título e palavras-chave em Português.

AGRADECIMENTOS

O Apoio financeiro de agências governamentais deve ser mencionado. Agradecimentos a auxílio técnico e assistência de colaboradores podem ser feitos neste capítulo.

REFERÊNCIAS

As referências devem ser apresentadas de acordo com o estilo do Brazilian Dental Journal. É recomendado aos autores consultar números recentes do BDJ para se familiarizar com a forma de citação das referências.

As referências devem ser numeradas por ordem de aparecimento no texto e citadas entre parênteses, sem espaço entre os números: (1), (3,5,8), (10-15). NÃO USAR SOBRESCRITO.

Para artigos com dois autores deve-se citar os dois nomes sempre que o artigo for referido. Ex: "According to Santos and Silva (1)...". Para artigos com três ou mais autores, citar apenas o primeiro autor, seguido de "et al.". Ex: "Pécora et al. (2) reported that..."

Na lista de referências, os nomes de TODOS OS AUTORES de cada artigo devem ser relacionados. Para trabalhos com 7 ou mais autores, os 6 primeiros autores devem ser listados seguido de "et al."

A lista de referências deve ser digitada no final do manuscrito, em seqüência numérica. Citar NO MÁXIMO 25 referências.

A citação de abstracts e livros, bem como de artigos publicados em revistas não indexadas deve ser evitada, a menos que seja absolutamente necessário. Não citar referências em Português.

Os títulos dos periódicos devem estar abreviados de acordo com o Dental Index. O estilo e pontuação das referências devem seguir o formato indicado abaixo:

Periódico

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.

Livro

2. Shafer WG, Hine MK, Levy BM. A textbook of oral pathology. 4th ed. Philadelphia: WB Saunders; 1983.

Capítulo de Livro

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.

TABELAS

As tabelas com seus respectivos títulos devem ser inseridas após o texto, numeradas com algarismos arábicos; NÃO UTILIZAR linhas verticais, negrito e letras maiúsculas (exceto as iniciais).

O título de cada tabela deve ser colocado na parte superior.

Cada tabela deve conter toda a informação necessária, de modo a ser compreendida independentemente do texto.

FIGURAS

NÃO SERÃO ACEITAS FIGURAS INSERIDAS EM ARQUIVOS ORIGINADOS EM EDITORES DE TEXTO COMO O WORD E NEM FIGURAS EM POWER POINT;

Os arquivos digitais das imagens devem ser gerados em Photoshop, Corel ou outro software similar, com extensão TIFF e resolução mínima de 300 dpi. Apenas figuras em PRETO E BRANCO são publicadas. Salvar as figuras no CD-ROM.

Letras e marcas de identificação devem ser claras e definidas. Áreas críticas de radiografias e fotomicrografias devem estar isoladas e/ou demarcadas.

Partes separadas de uma mesma figura devem ser legendadas com letras maiúsculas (A, B, C, etc). Figuras simples e pranchas de figuras devem ter largura mínima de 8 cm e 16 cm, respectivamente.

As legendas das figuras devem ser numeradas com algarismos arábicos e apresentadas em uma página separada, após a lista de referências (ou após as tabelas, quando houver).

Não há taxas para submissão e avaliação de artigos.

A Taxa de Revisão técnica varia de R\$ 450,00 a R\$ 500,00 Reais Brasileiros (para autores nacionais) ou U\$ 200 a U\$ 300 Dólares Americanos (para autores estrangeiros) e será cobrada do autor correspondente, ainda que apenas pequenas correções no manuscrito sejam necessárias.

NORMAS PARA PUBLICAÇÃO NO PERIÓDICO BRAZILIAN ORAL RESEARCH

Mission, scope, and submission policy

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 journal has an Impact Factor™ of 0.937 (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.

BOR invites the submission of original and review manuscripts and papers in the following typology: Original Research (complete manuscript or Short Communication), Critical Review of Literature, Systematic Review (and Meta-Analysis) and Letters to the Editor. All submissions must be exclusive to.

Manuscripts and all corresponding documentation should be exclusively submitted through ScholarOne Manuscripts™ via the online submission link (<http://mc04.manuscriptcentral.com/bor-scielo>).

The evaluation process of manuscript's scientific content will only be initiated after meeting of all the requirements described in the present Instructions for Authors. Any manuscript that does not meet these requirements will be returned to the corresponding author for adaptations.

Important: Once having been accepted on their scientific merit, all manuscripts will be submitted for grammar and style revision as per the English language. Contact BOR by bor@sbpqo.org.br to get information about the recommended translation companies. The authors should forward the revised text with the enclosed revision certificate provided by the chosen editing company. Linguistic revisions performed by companies that do not provide the mentioned certificate will not be accepted. As an exception, this rule does not apply when one of the authors is a native English speaker.

Presentation of the manuscript

The manuscript text should be written in English and provided in a digital file compatible with "Microsoft Word" (in DOC, DOCX, or RTF format).

All figures (including those in layouts/combinations) must be provided in individual and separate files, according to recommendations described under the specific topic.

Photographs, micrographs, and radiographs should be provided in TIFF format, according to the recommendations described under the specific topic.

Charts, drawings, layouts, and other vector illustrations must be provided in a PDF format individually in separate files, according to the recommendations described under the specific topic.

Video files may be submitted as per the specifications, including the author's anonymity (for purposes of evaluation) and respect for the patient's rights.

Important: ScholarOne™ allows upload of a set of files up to 10 MB. In case the video file exceeds this size, it is possible to leave information about the link to access the video. The use of patients' initials, names, and/or registry numbers is prohibited in the reproduction of clinical documentation. The identification of patients is prohibited. An informed consent statement, signed by the patient, concerning the use of his/her image should be provided by the author(s) when requested by BOR. The Copyright legislation in force must be respected and the source cited when the manuscript reproduces any previously published material (including texts, charts, tables, figures, or any other materials).

Title page (compulsory data)

This must indicate the specialty* or research field focused on in the manuscript.

*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.

Informative and concise title, limited to a maximum of 110 characters, including spaces.

Names of all authors written out in full, including respective telephone numbers and email addresses for correspondence. We recommend that authors collate the names present in the Cover Letter with the profile created in ScholarOne™, to avoid discrepancies.

The participation of each author must be justified on a separate page, which should meet the authorship and co-authorship criteria adopted by the

International Committee of Medical Journal Editors, available at <http://www.icmje.org/recommendations/browse/roles-and-responsibilities/defining-the-role-of-authors-and-contributors.html>

Data of institutional/professional affiliation of all authors, including university (or other institution), college/program, department, city, state, and country, presented according to internal citation norms established by each author's institution. Verify that such affiliations are correctly entered in ScholarOne™.

Abstract: This should be presented as a single structured paragraph (but with no subdivisions into sections) containing the objective of the work, 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 <http://decs.bvs.br/> or <http://www.nlm.nih.gov/mesh/MBrowser.html> (no synonyms will be accepted).

Main Text

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://stroke-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/>).

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: These 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 RTF format.

Discussion: This must 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: These 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.

Plagiarism

BOR employs a plagiarism detection system. When you send your manuscript to the journal it may be analyzed-not merely for the repetition of names/affiliations, but rather the sentences or texts used.

References: Only publications from peer-reviewed journals will be accepted as references. Unfinished manuscripts, dissertations, theses, or abstracts presented in congresses will not be accepted as references. References to books should be avoided.

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 (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.

Characteristics and layouts of types of manuscripts

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 - Text Files

- 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
- Tables
- References: maximum of 40 references

- Figure legends
- Layout - Graphic Files
- Figures: a maximum of 8 (eight) figures, as described above.

Copyright transfer agreement and responsibility statements

The manuscript submitted for publication must include the Copyright Transfer Agreement and the Responsibility Statements, available in the online system and mandatory.

EXAMPLES OF REFERENCES

Journals

Goracci C, Tavares AU, Fabianelli A, Monticelli F, Raffaelli O, Cardoso PC, et al. The adhesion between fiber posts and root canal walls: comparison between microtensile and push-out bond strength measurements. *Eur J Oral Sci.* 2004 Aug;112(4):353-61.

Walsh B, Steiner A, Pickering RM, Ward-Basu J. Economic evaluation of nurse led intermediate care versus standard care for post-acute medical patients: cost minimisation analysis of data from a randomised controlled trial. *BMJ.* 2005 Mar 26;330(7493):699. Epub 2005 Mar 9.

Papers with Title and Text in Languages 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.

Supplements or Special Editions

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.

Online Journals

Barata RB, Ribeiro MCSA, De Sordi M. Desigualdades sociais e homicídios na cidade de São Paulo, 1998. Rev Bras Epidemiol. 2008;11(1):3-13 [cited 2008 Feb 23]. Available from: <http://www.scielo.org/pdf/rbepid/v11n1/01.pdf>.

Books

Stedman TL. Stedman's medical dictionary: a vocabulary of medicine and its allied sciences, with pronunciations and derivations. 20th ed. Baltimore: Williams & Wilkins; 1961. 259 p.

Books Online

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/>.

Websites

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/>.

World Health Organization [homepage]. Geneva: World Health Organization; 2011 [cited 2011 Jan 17]. Available from: <http://www.who.int/en/>

PRODUÇÃO CIENTÍFICA (2014-2018)

Artigos completos

* Viability of bovine teeth as a substrate in bond strength tests: systematic review and meta-analysis

Short title: Bovine teeth: viability in bond strength tests.

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Nayara Kelly Lyrio Ferraza^a, Allyson Moreira Nogueira^c, Mônica Yamauti^d,
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Abstract

Purpose: A systematic review and meta-analysis of the scientific data comparing bovine and human permanent and deciduous teeth used for tensile and shear bond strength tests. **Material and Methods:** Articles were selected from Web of Science, PubMed, Scopus, LILACS-Bireme and BBO electronic databases using keywords obtained from Medical Subject Headings (MeSH). Of 1304 potentially eligible studies, 155 were selected for full text analysis. Five independent reviewers ($Kappa=0.89$) selected the studies, abstracted information and assessed quality based on standardized scales. After the analysis, 76 studies comparing bovine teeth to human teeth were found. Only 19 studies comparing bovine and human substrates in bond strength tests were included in the systematic review and 14 in the meta-analysis. Two authors independently selected the studies, extracted the data and assessed the risk of bias. Mean differences were obtained by comparing tensile and shear bond strength values between human and bovine teeth (permanent and deciduous) and considering enamel and dentin separately (subgroup analysis). Statistical analysis was performed using RevMan5.1, with a random-effect model, at a 5% significance level.

Results: No significant difference was found between human and bovine teeth in tensile tests ($p=0.41$) for dentin ($p=0.86$), but there was a difference for enamel ($p=0.01$). Regarding shear bond strength, no significant difference was found between human and bovine teeth ($p=0.16$) either for enamel ($p=0.07$) or for dentin ($p=0.68$) substrates. Regarding shear bond strength on deciduous teeth, no significant difference was found between human and bovine substrates ($p=0.54$), either for enamel ($p=0.42$) or for dentin ($p=0.05$). All included studies in the systematic review scored between a low and high risk of bias.

Conclusions: Bovine teeth can be a suitable alternative for permanent and deciduous human teeth, both for enamel and dentin substrates, with respect to tensile and shear bond strength.

Keywords: Dentin; Enamel; Human tooth; Bovine tooth; Bond strength.

INTRODUCTION

Dental adhesive systems provide deep modifications in restorative procedures, making them more conservative and durable. The evolution of tooth adhesion and the development of dental materials first involved laboratory studies.¹ *In vitro* studies initially used human teeth, which become more difficult to obtain due to a conservative dentistry approach.¹⁴⁻²² In addition, ethical aspects demand attention when human teeth are involved in *in vitro* studies.^{18,26}

Bovine teeth have been used in laboratory research as an alternative substrate to human teeth, mainly because of the ready availability, the absence of carious lesions, and a size that facilitates the technical procedures.^{22,25} Bovine teeth have also been used as an alternative substrate in bond strength tests.^{10,18} Different studies have evaluated both substrates, aiming to establish the bovine tooth as a human substitute.^{1,3,10,9,22} However, these studies vary widely in their materials and methods, raising some concern about the application of data obtained from bovine teeth to the clinical situation.^{2,10}

A pooled analysis of nine bond strength studies with meta-analyses was recently published. The results indicated that bovine teeth produce results similar to those of human teeth.²⁶ However, an additional analysis for the different types of bond strength tests is recommended. It is also important to select studies with methodological consistency in order to develop a consensus on this question. The present investigation aimed to update the subject and conduct a systematic review and meta-analysis comparing the results of different bond strength tests to answer the following question: Do bovine teeth substitute for human teeth in tensile and shear bond strength tests? The null hypothesis tested was that bovine teeth do not differ from human teeth as a substrate for tensile and shear bond strength tests.

MATERIALS AND METHODS

This study was conducted according to the PRISMA Statement criteria for systematic reviews and meta-analyses.¹² The following research question was formulated to add to the literature and to outline the search strategy: For which property tests can the bovine tooth replace the human tooth in laboratory studies?

Search strategy and selection criteria

Web of Science, PubMed, Scopus, LILACS (Bireme) and BBO databases were searched to identify studies comparing the use of human and bovine teeth. Original articles published until September 17, 2015, in the Portuguese, English and Spanish

languages were included without a date restriction. The search was updated on April 04, 2018.

The search strategies included keywords obtained from *Medical Subject Headings* (*MeSH*) and were appropriately modified for each database. In PubMed and Web of Science, the following strategy was used: ("Dentin"[Mesh] AND Dentin OR "Dental Enamel"[Mesh] AND enamel) AND ("Tooth Root"[Mesh] OR "Human tooth" OR human tooth OR tooth OR teeth) AND ("bovine tooth" OR bovine tooth OR "bovine teeth" OR bovine teeth). For Lilacs (Bireme), BBO and Scopus, short combinations were made using the Boolean operators and/or, with the following terms: *dentin*, *dental enamel*, *tooth root*, *human tooth*, *tooth*, *teeth*, *bovine tooth*, *bovine teeth*.

The following inclusion criteria were used for the selection of studies: (1) laboratory studies and (2) studies that presented a comparison between human and bovine teeth. There was no restriction regarding the chronological age of the teeth, inclusion of teeth of other animal species, or time and means of storage. Exclusion criteria eliminated descriptive articles that did not compare human and bovine teeth, review articles, and studies published on other languages rather than English, Spanish and Portuguese. Academic theses were also excluded. No period restriction was applied.

The eligibility of the studies was determined by reading the titles and abstracts of the articles identified in each database. Articles presenting a comparison between bovine teeth and human teeth in the title and abstract were selected to read in full. The data retrieved were imported by EndNote software, and a final list, after excluding duplicates, was analyzed by five calibrated researchers (ACNL, LCN, MCNSR, MFEC, NKLF). For the calibration exercise, the researchers discussed the criteria and applied them in a sample of 10% of the recovered studies. Calibration was performed to obtain excellent interexaminer agreement ($Kappa=0.89$). Each study was independently reviewed, and classifications were compared. Interexaminer divergences were discussed until reaching a consensus.

Data collection

In the search, there was no restriction on the property evaluated. Due to the large volume of articles found, it was decided to split the results into two articles: the descriptive article was developed to evaluate different parameters such as radiodensity, microleakage, surface roughness, compression, extension, tensile properties, erosion, abrasion, etc., and contained 78 articles; for this study, 18 articles included a meta-analysis regarding the bond strength. A standard spreadsheet was created in Microsoft

Office Excel 2013 (Microsoft Corporation, Redmond, WA, EUA) to extract data from the articles and classified according to the quality of research (Table 1). The following data were systematically extracted from each article: authors; year of publication; article title; journal title, volume and page; language; study location; objective; response variable; laboratory test; dental substrate (enamel, dentin, cement); sample size; storage and disinfection medium; evaluated groups; preparation of specimens; thermal cycling; storage time and conditions until evaluation; statistical analysis; results (p value, mean, standard deviation) and conclusions.

Statistical analysis

For the meta-analyses, three distinct data analyses were carried out: 1) human vs. bovine teeth in a tensile bond strength test, 2) human vs. bovine teeth in a shear bond strength test and 3) human deciduous vs. bovine deciduous teeth in a bond strength test. The pooled effect estimates were obtained by comparing means to measure the possibility to compare human and bovine teeth, considering the subgroups and type of substrate (enamel or dentin). For the studies that evaluated different materials or depths of dentin, the values were extracted to calculate the mean and standard deviation for human and bovine groups. In the selected studies, only the data of interest were extracted to be analyzed in the meta-analysis. If the study performed a separate group with no etching for the adhesive system, the group was excluded from the analysis. For longevity studies that had storage groups for aging, only the initial results of bond strength were considered, independently of the evaluation time.

The significant differences between groups were calculated in Review Manager (RevMan version 5.3 software, Cochrane Collaboration, Copenhagen, Denmark, 2014) using a random-effect method. Statistical significance was defined as $p\text{-value} \leq 0.05$ (Z test). Forest plots were created to illustrate the meta-analysis. Heterogeneity between studies was assessed by Cochran's Q test with a significance level of 0.10. The I^2 statistic was also examined, considering $I^2 > 60\%$ to indicate significant heterogeneity.

RESULTS

Search and selection

The search results are shown in the flow diagram (Figure 1). A total of 1540 potentially eligible studies were found. After duplicate articles were disregarded, 1481 were selected for abstract review; 78 studies met the inclusion criteria for further assessment, and 24 met the inclusion criteria for bond strength tests. After analysis, 5 articles were excluded because human and bovine teeth were not compared^{15,17,28} or

different substrates were compared.^{23,29} As two studies were included from the crossed references,^{20,19} 18 studies were included in the systematic review. However, 6 studies were not included in the meta-analyses: 2 of them were excluded because of the type of test evaluated (fracture resistance test; push-out test),^{7,25} 1 was not included because of the absence of numerical data,²⁴ 1 was excluded because of the substrate evaluated,³² and 1 was excluded because it was a systematic review.²⁶

Descriptive analysis

Nineteen articles fulfilled the inclusion criteria for systematic review and were evaluated and classified according to the quality of research, with a score ranging from 3 to 4 (Table 2). The methodological characteristics of the studies are shown in Table 3. All studies were published between 1983 and 2018. The majority of the studies conducted the bond strength test after 24 hours;^{1,3,11,14,15,18-22} shear bond strength was the most used test, followed by the tensile bond strength test. Moreover, 8 studies compared both enamel and dentin in human and bovine permanent teeth,^{3,6,11,14,18,20,21,28} and 2 studies compared enamel and dentin in human and bovine deciduous teeth.^{9,31} Only 1 study compared human and bovine substrates in the dentin-enamel junction.³² One study performed a *push-out* design for evaluating the bond strength to root dentin,⁷ and one employed a variation of the shear bond test using a fracture resistance test.²⁵

Meta-analyses

The meta-analyses were performed considering the type of teeth (permanent or deciduous) and the type of test (tensile or shear bond strength) and considering two subgroup analyses (enamel and dentin, separately). Of the 14 studies included in the meta-analyses, 2 studies evaluated deciduous teeth with respect to shear bond strength,^{9,31} and only 1 study evaluated both types of tests.⁶

Results of the meta-analyses are presented in Figures 2, 3 and 4. The analysis between human and bovine teeth with respect to tensile bond test (Figure 2) did not find a difference between groups ($p=0.41$), with moderate heterogeneity ($I^2=36\%$). The subgroup meta-analysis showed that human enamel produced lower tensile bond strength values than did bovine enamel ($p=0.01$), with $I^2=0\%$. When the values for dentin were analyzed, there was not a significant difference between the human and bovine substrate ($p=0.86$), with $I^2=18\%$. The global analysis for shear bond strength of human and bovine teeth (Figure 3) did not reveal a difference between groups ($p=0.16$), with $I^2=79\%$. For the subgroup meta-analysis, no significant differences were found for both enamel ($p=0.07$) and dentin ($p=0.68$), with moderate and high heterogeneity, $I^2=56\%$ and

$I^2=85\%$, respectively. Regarding the shear bond strength of human and bovine deciduous teeth (Figure 4), the global analysis showed no difference ($p=0.54$), with $I^2=84\%$. Indeed, there was no significant difference between the enamel ($p=0.42$, with $I^2=37\%$) and dentin ($p=0.05$, with $I^2=50\%$) subgroups.

DISCUSSION

This systematic review involved the search for articles comparing the use of bovine and human teeth for *in vitro* tests. Due to differences in the methods found, the authors decided to divide the study into two reviews to facilitate the analysis. For this study, specifically, the systematic review summarizes the use of bovine teeth as substitutes of human teeth in bond strength tests. The global analysis clarified the viability for using bovine teeth for bond strength tests and supported the results of laboratory studies. The null hypothesis stating that bovine teeth do not differ from human teeth in bond strength tests was accepted.

For this systematic review, 18 *in vitro* studies were selected, and 14 were also included in the meta-analysis. Incomplete data and different substrates and tests were the reasons for the exclusion of 5 articles from the meta-analysis.^{7,25,26,24,32} Our global-analysis results showed no difference in bond strength mean values (for tensile and shear bonding) between bovine and human teeth, for both enamel vs. dentin and permanent teeth vs. deciduous teeth.

The number of studies included in the meta-analysis of tensile test for the enamel^{6,14,18} and dentin^{6,8,13,14,18} subgroups was different. The overall effect was not significant for the type of substrate used. Moreover, the heterogeneity found for enamel ($I^2=0\%$; $p=0.73$), dentin ($I^2=18\%$; $p=0.30$) and the global analysis ($I^2=36\%$, $p=0.14$) might not be relevant since the statistical test did not provide evidence on this effect. Otherwise, substantial heterogeneity was found for shear bond strength^{1,3,6,10,19,20,22} of the dentin subgroup ($I^2=85\%$, $p<0.001$) and the overall effect ($I^2=79\%$; $p<0.001$). This heterogeneity is probably because more than eight adhesive systems were used in the different studies. Indeed, for the shear bond strength of deciduous teeth,^{9,31} only the global analysis ($I^2=84\%$, $p=0.0003$) revealed a substantial heterogeneity, most likely because of the reduced number of studies in this subgroup.

It is well known that human dental enamel is composed of 96% of inorganic matrix and 4% of organic material, which comprises lipids, proteins and water.⁵ Human enamel shows the highest mineralization degree, and in this substrate, calcium and

phosphorus were the most frequently found chemical elements, with bovine teeth demonstrating the most similar Ca/P ratios.²⁹ However, structural differences, such as density and size of crystals should be responsible for the significant difference in enamel between human and bovine teeth in tensile tests,³⁰ since the physical and mechanical properties of teeth depend on not only the composition but also the structure of the substrate.¹⁶

Differences in morphological and structural characteristics between permanent and primary teeth can be assumed by the higher tubule density and larger tubule diameters in primary dentin.⁹ Our results for deciduous teeth in the dentin substrate found a borderline result ($p=0.05$). This could be explained for the dentin morphological and chemical features. Unfortunately, descriptions of element concentrations in the literature regarding deciduous teeth are scarce.²⁷

Independently of the type of test analyzed (tensile or shear bond test), there was no significant difference between the substrates. Our results are in accordance with a previous systematic review for bond strength published in 2016.²⁶ However, it is necessary to evaluate the results with caution, due to the lower number of studies with similar methodologies and sample sizes.

All studies included in this systematic review exhibited a moderate risk of bias. The quality assessment followed a guideline and checklist for the methodological development of *in vitro* studies.⁴ Useful information about sample size, randomizing and blinding were not often available in the assessed studies. Therefore, the careful design of future *in vitro* studies is recommended.

CONCLUSION

The available scientific data indicates the viability of bovine teeth to be used as an alternative for human permanent and deciduous teeth, for both enamel- and dentin-substrate shear bond strength tests. However, the tensile bond strength of enamel needs attention, as it can be observed that bovine enamel reduces the capacity of a dental structure in this type of bond test.

Clinical Relevance: For the necessity to find a substitute for human teeth to bond strength test. Bovine teeth can be a suitable alternative for tensile and shear bond strength.

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Figures

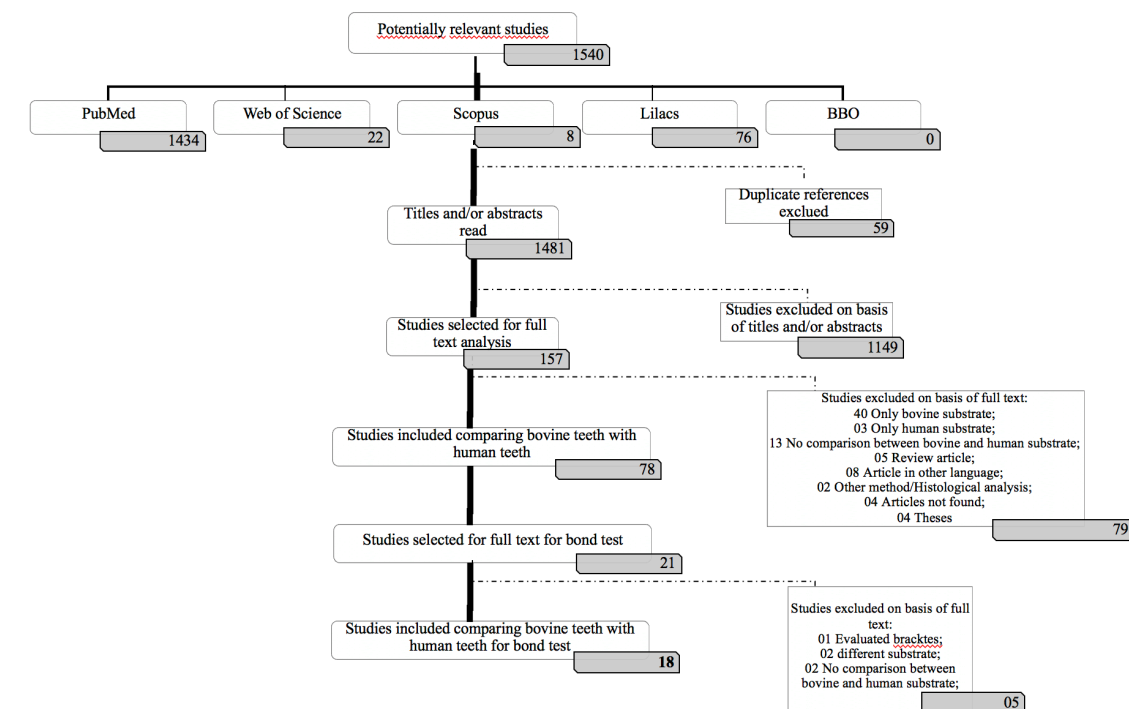


Figure 1. Flow diagram showing the process of selecting published articles.

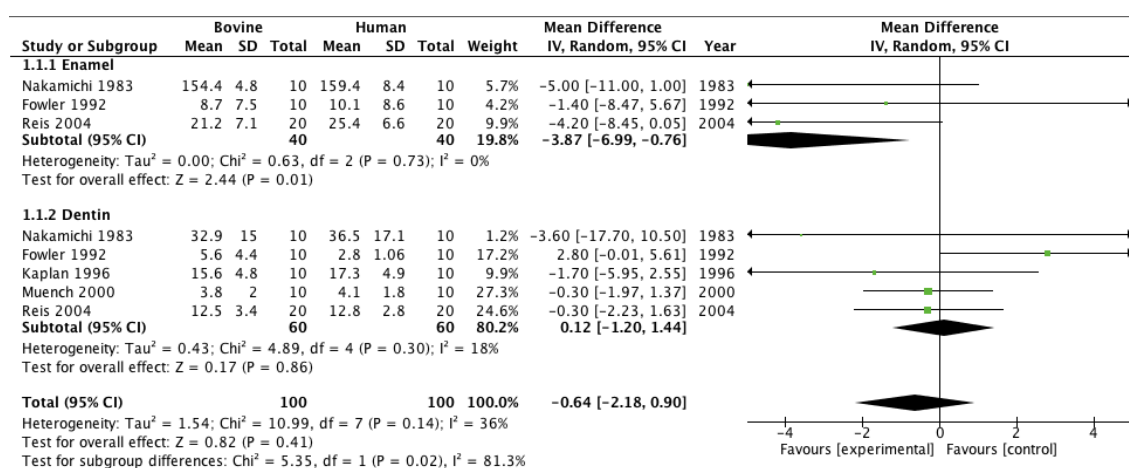


Figure 2. Forest plot for the analysis of tensile bond test between bovine and human substrates.

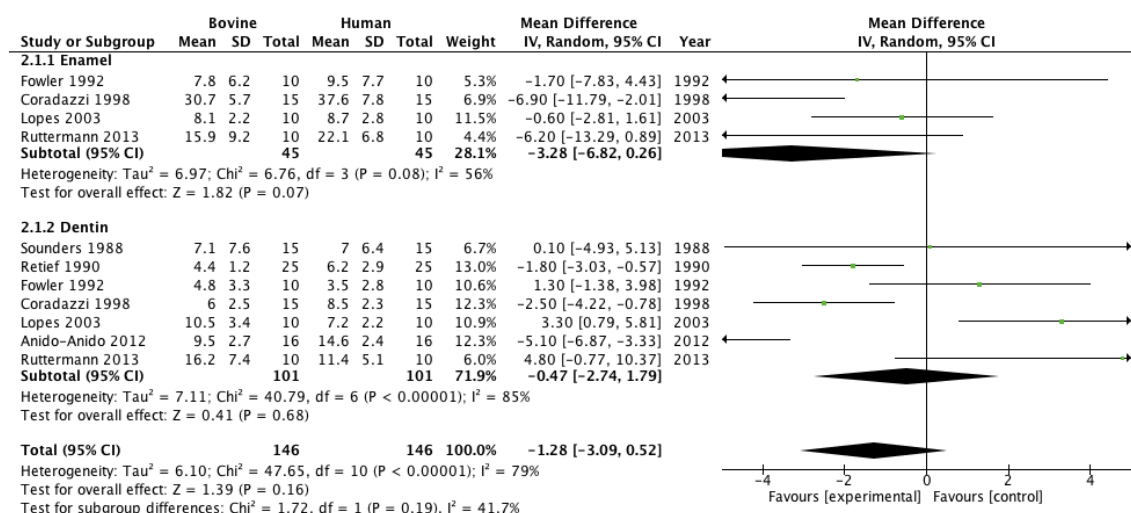


Figure 3. Forest plot for the analysis of shear bond strength between bovine and human substrates.

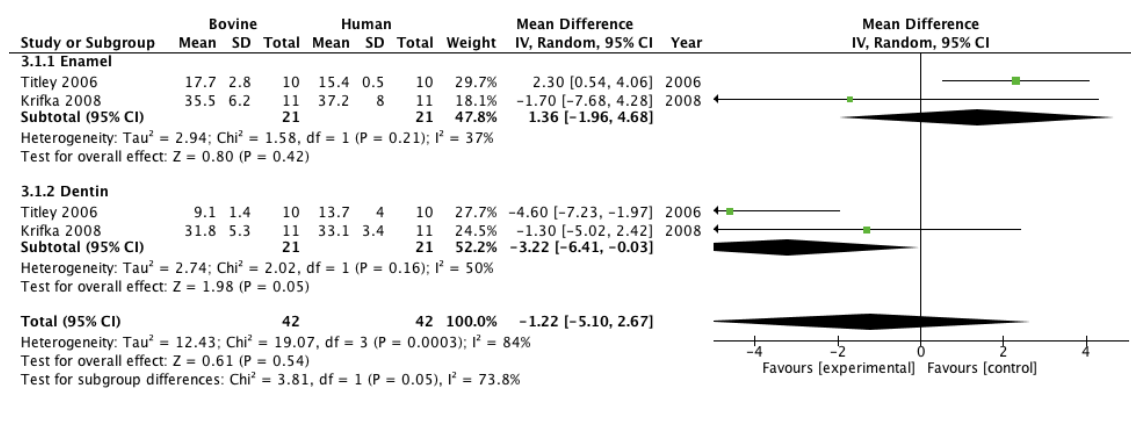


Figure 4. Forest plot for the analysis of tensile bond test between bovine deciduous and human deciduous substrate.

Table 1. Description of criteria for evaluating the quality of the selected articles.

Criteria	Classification	Score	Definition
1. Intervention	Adequate	1.0	The intervention for each group, including how and when it was administered, with sufficient detail to enable replication
	Inadequate	0.5	Data not clearly presented in the article
	Not described	0.0	Insufficient data
2. Outcomes	Adequate	1.0	Completely defined, pre-specified primary and secondary measures of outcome, including how and when they were assessed
	Inadequate	0.5	Insufficient data
	Not described	0.0	Data not clearly presented in the article
3. Sample size	Adequate	1.0	How sample size was determined
	Not described	0.0	Does not describe/present/communicate conducting sample calculation
4. Randomization	Adequate	1.0	Method used to generate and to implement the random allocation sequence, describing any steps taken to conceal the sequence until intervention was assigned
	Inadequate	0.5	Insufficient data
	Not described	0.0	Data not clearly presented in the article
5. Implementation	Adequate	1.0	Who generated the random allocation sequence, who enrolled teeth, and who assigned teeth to intervention
	Inadequate	0.5	Insufficient data
	Not described	0.0	Data not clearly presented in the article
6. Blinding	Adequate	1.0	If done, who was blinded after assignment to intervention and how
	Inadequate	0.5	Insufficient data
	Not described	0.0	Data not clearly presented in the article
7. Statistical methods	Adequate	1.0	Statistical treatment fully described and adequate
	Inadequate	0.5	Statistical treatment not fully described and inadequate
	Not described	0.0	No statistical treatment applied

Checklist/Scale adapted for this study from Faggion Jr (2012)

Table 2. Quality assessment of the selected studies, research quality or methodological soundness: high (≥ 5), moderate (3 - 5) or low (< 3).

Article	Intervention	Outcomes	Sample size	Randomization	Implementation	Blinding	Statistical methods	Total points	Research quality
Nakamichi <i>et al.</i> , 1983 ¹⁴	1	1	0	0	0	0	1	3	Moderate
Saunders, 1988 ²²	1	1	0	1	0	0	1	4	Moderate
Retief, 1990 ¹⁹	1	1	0	0	0	0	1	3	Moderate
Fowler <i>et al.</i> , 1992 ⁶	1	1	0	0	0	0	1	3	Moderate
Kaplan & Ubios & Beigelis, 1996 ⁸	1	1	0	0	0	0	1	3	Moderate
Shahabi, Brockhurst & Walsh, 1997 ²⁴	1	1	0	0,5	0	0	1	3,5	Moderate
Coradazzi <i>et al.</i> , 1998 ³	1	1	0	0,5	0	0	1	3,5	Moderate
Muench & da Silva & Ballester, 2000 ¹³	1	1	0	1	0	0	0	3	Moderate
Urabe <i>et al.</i> , 2000 ³²	1	1	0	0	0	0	1	3	Moderate
Lopes <i>et al.</i> , 2003 ¹¹	1	1	0	0	0	0	1	3	Moderate
Reis <i>et al.</i> , 2004 ¹⁸	1	1	0	0,5	0	0	1	3,5	Moderate
Titley & Childers & Kulkarni, 2006 ³¹	1	1	0	0	0	0	1	3	Moderate
Krifka <i>et al.</i> , 2008 ⁹	1	1	0	0,5	0	0	1	3,5	Moderate
Galhano <i>et al.</i> , 2009 ⁷	1	1	0	1	0	0	1	3	Moderate
Soares <i>et al.</i> , 2010 ²⁵	1	1	0	0	0	0	1	3	Moderate
Anido-Anido <i>et al.</i> , 2012 ¹	1	1	0	0	0	0	1	3	Moderate
Ruttermann, Braun & Jand, 2013 ²⁰	1	1	0	0,5	0	0	0,5	3,5	Moderate
Soares <i>et al.</i> , 2016 ²⁶	1	1	1	0	0	0	1	4	Moderate

Table 3. Description data from included studies according to substrate, comparison, type of test and conclusion.

Author, Year	Substrate	Comparison	Bond Strength Test	Conclusion
Nakamichi <i>et al.</i> , 1983 ¹⁴	Enamel and dentin human and bovine teeth	Bond strength of different materials (Polycarboxylate cement; Glass-ionomer cement; Zinc phosphate cement; Composite resin) in different substrates of human and bovine teeth	Tensile	Bovine teeth were useful in the adhesion test as substitutes for human teeth using enamel and the superficial layer of dentin
Saunders, 1988 ²²	Dentin human and bovine teeth	Bond strength of different materials (Scotchbond; Topaz; Gluma; 3M experimental agent) in superficial layer of dentin, after 24 hours	Shear	Bovine specimens can be used as a suitable substitute of human teeth in shear bond strength studies utilizing superficial layer of dentin
Retief, 1990 ¹⁹	Dentin human and bovine teeth	Bond strength of two materials (Schotchbond 2; Silux) in dentin of human and bovine teeth, after 24 hours	Shear	The use of bovine teeth instead of human teeth in these types of tests is not indicated.
Fowler <i>et al.</i> , 1992 ⁶	Enamel and dentin human and bovine teeth	Bond strength of two materials (Schotchbond; Silux; Ketac – Fil; Vitrebond) in enamel and dentin of human and bovine teeth, for 2 weeks	Tensile and Shear	Bond strength obtained with human and bovine enamel were essentially comparable. Values with dentin varied. There appeared to be a trend for higher bond strength values with bovine than with human dentin
Kaplan & Ubios & Beigelis, 1996 ⁸	Dentin human and bovine teeth	Bond strength of adhesive systems (Scotchbond, Bondlite, Prisma Universal Bond, Gluma) in dentin surface, after 7 days	Tensile	Results showed that more reliable values were obtained when using bovine dentin
Shahabi & Brockhurst & Walsh, 1997 ²⁴	Enamel human, porcine and bovine teeth	Shear bond strength (Schotchbond multi-purpose) of composite resin to enamel to of human, porcine and bovine teeth, after 48 hours	Shear	Porcine and bovine enamel behaves similarly to human enamel in terms of both acid etching and laser conditioning for bond strength
Coradazzi <i>et al.</i> , 1998 ³	Enamel and dentin human, swinish and bovine teeth	Bond strength of adhesive system (Prisma Universal Bond 3) in enamel and dentin surfaces, after 24 hours and 7 days	Shear	The shear bond strength in human enamel present not statistically significant difference of bovine enamel, but differences to swinish enamel and in dentin substrate any significant difference was present
Muench & da Silva & Ballester, 2000 ¹³	Dentin human and bovine teeth	Bond strength of adhesive systems (Prime&Bond, Single Bond, Etch&Prime) in dentin surface	Tensile	The differences among the dentin substrates do not appear to be critical for the tensile bond strength test for the three adhesive systems
Urabe <i>et al.</i> , 2000 ³²	Dentin-enamel junction human and bovine teeth	Bond strength of adhesive system (Clearfil PhotoBond) after 24 hours	Microtensile Test	There was no difference between dentin-enamel junction for tensile strength when compared human and bovine teeth
Lopes <i>et al.</i> , 2003 ¹¹	Enamel and dentin human and bovine teeth	Bond strength of adhesive systems (Scotchbond Multi-Purpose; Clearfil Liner Bond 2V) in enamel and dentin surfaces, after 24 hours	Shear	The use of bovine teeth in place of human teeth in laboratory tests of shear bond strength seems to be partially valid because the obtained values were not always proportional to the two substrates; this depends on the adhesive system used

Reis <i>et al.</i> , 2004 ¹⁸	Enamel and dentin human, porcine and bovine teeth	Bond strength of adhesive system (Single bond) in enamel and dentin surfaces at 24 hours	Microtensile	It can be concluded that although human teeth are preferable, bovine teeth are better substitutes than porcine teeth in testing resin adhesion.
Titley & Childers & Kulkarni, 2006 ³¹	Enamel and dentin deciduous human and bovine teeth	Bond strength of adhesive systems (F2000 one-step primer/adhesive; Prime and Bond NT) in enamel and dentin surfaces, after 7 and 180 days	Shear	Bovine primary incisor enamel and dentine provided the latter is not conditioned with 37% orthophosphoric acid, are suitable alternative test substrates for human primary enamel and dentine
Krifka <i>et al.</i> , 2008 ⁹	Enamel and dentin deciduous human and bovine teeth	Bond strength of adhesive systems (Syntac Assortment; Adaper Prompt L-Pop; iBond Gluma; Clearfil Protect Bond) in enamel and dentin surfaces, after 24 hours	Shear	Bovine primary teeth hard tissue substrates may be regarded as suitable alternatives to teeth of human origin for shear bond testing
Galhano <i>et al.</i> , 2009 ⁷	Root dentin human and bovine teeth	Bond strength of adhesive (All Bond 2) to human and bovine dentin root, after 24 hours	Push-out	The push-out strengths of bovine- and human-root dentin were statistically different
Soares <i>et al.</i> , 2010 ²⁵	Enamel human and bovine teeth	Determine influence of fiber reinforcement on fracture strength of composite partial denture	Fracture Resistance	Bovine teeth can be used as substitutes for human teeth in these types of fracture strength tests
Anido-Anido <i>et al.</i> , 2012 ¹	Dentin human and bovine teeth	Bond strength of adhesive system (Scotchbond Multi-Purpose Adhesive) to bovine and human dentin, in different depths, after 24 hours	Shear	Bovine substrate can be used for laboratory studies of bond strength as indicative of the initial performance of new products, although the differences related to human substrate should be observed
Ruttermann, Braun & Jand, 2013 ²⁰	Enamel and dentin human and bovine teeth	Bond strengths of adhesive system (Clearfil SE Bond; Optibond FL) in enamel and dentin surfaces, after 24 hours and 500 termocycles	Shear	Bovine teeth can only partly be recommended as a substitute for human teeth
Soares <i>et al.</i> , 2016 ²⁶	Enamel and dentin human and bovine teeth	Bond strengths in enamel and dentin surfaces	Tensile and Shear	Bovine teeth can be considered as a suitable substitute for human teeth on bond strength tests.

Title Page

**Ethanol wet-bonding application on bond strength in root canal: A
systematic review**

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Abstract

Purpose: A systematic review and meta-analysis were performed to answer the following research question: Can ethanol wet-bonding be an alternative to water wet-bonding for the bond strength and durability of adhesion of fiber posts in laboratory studies?

Material and Methods: *in vitro* study that involving root canal with ethanol wet-bonding and water wet-bonding were included. A comprehensive search for titles and abstracts using keywords obtained from the Medical Subject Headings (MeSH) was performed without restrictions. The use of random effects model, a meta-analysis was conducted for time of storage (24 hours and 12 months) with a subgroup analysis (etch-and-rise and self-etching adhesive system) for 24 hours.

Results: We retrieved 1271 articles, after removing duplicates and non-relevant articles, only 26 *in vitro* studies remained. No significant difference in ethanol wet-bonding and water wet-bonding in 24 hours was observed for different adhesive system ($p>0.05$). For etch-and-rise adhesive system after 12 months' water wet-bonding were significantly different ($p<0.05$).

Conclusion: The use of water wet-bonding with etch-and-rise adhesive system for *in vitro* study seem to improve the bond strength after 12 months.

KEYWORDS: Adhesion; Root dentin; Ethanol wet-bonding; Bond strength; Bond durability.

INTRODUCTION

The restoration of endodontically treated teeth using fiber posts has increased in daily practice.^{4,39} The clinical success depends on endodontic treatment, the method of cement application, post pretreatment⁴⁴ and, mainly, a reliable bond at the post/dentin interface.¹³ However, the main reason for failure of fiber posts is still debonding,³⁹ which is the most common failure from a decrease in retention at the dentin/cement interface.³⁶

Different studies have correlated the instability of the resin-dentin bond with the hydrophilic resin monomer in dentin adhesive^{24,26,34} that increases water sorption^{22,51} and decreases mechanical properties.^{24,37} In theory, hydrolysis of resin-dentin bonds involves the degradation of both components of hybridized dentin, resin and collagen fibrils.²⁶ The alternative to extend the longevity of a resin-dentin bond is to make acid-etched dentin less hydrophilic.⁴⁵

Ethanol wet-bonding has been proposed for Pashley et al., 2007³⁴ and was characterized by sequential rinses with ethanol at ascending concentrations to replace interfibrillar water.⁹ Theoretically, ethanol wet-bonding displaces all water present in acid-etched dentin with ethanol, leaving the unsupported collagen fibrils with ethanol and allowing the monomer resins to impregnate the substrate.³⁴ Despite this theory, a resin-dentin bond could preserve bond strength⁷ to reduce water sorption³⁸ and micropermeability.⁴⁰

This technique was initially developed for etch&rinse adhesive systems³⁴ to overcome the degradation of non-protected demineralized dentin.⁴⁸ Despite promising *in vitro* results, ethanol wet-bonding became to be associated with different materials as, self-adhesive system,^{2,11} self-adhesive resin cement^{3,4} and an experimental hydrophobic adhesive.^{9,35} Indeed, the time consuming of sequential rinses proposed a search for a reduction of time application.^{9,10,11}

Despite the number of *in vitro* studies in the literature, there is no strong evidence supporting that ethanol wet-bonding is able to improve the retention of fiber posts in root canals. Thus, the aim of this study was to systematically review the literature for *in vitro* studies that evaluated the use of simplified ethanol wet-bonding for the bond strength of root dentin. The hypothesis tested

was that ethanol wet-bonding does not improve the bond strength and durability of adhesion of fiber posts on root dentin.

MATERIALS AND METHODS

A systematic review was conducted of laboratory studies that provided the best synthesis of evidence on this topic. This type of review can also be called a “systematic search and review”, an adaptation of the *classic* systematic review for non-epidemiological studies.²¹

This study was conducted according to the Preferred Reporting Items for Systematic Reviews and Meta-analyses (PRISMA Statement) criteria for systematic reviews and meta-analysis.²⁸ The following research question was formulated to add to the literature and outline the search strategy: Can ethanol wet-bonding be an alternative to water wet-bonding for the bond strength and durability of adhesion of fiber posts in laboratory studies?

Search strategy and selection criteria

PubMed, Web of Science, Scopus, BBO, Lilacs (Bireme), and Medline via Ovid databases were searched to identify studies that compared the use of ethanol wet-bonding in root canals. Original articles published through August 03, 2017, without language or date restrictions, were included.

The search strategies included keywords obtained in the *Medical Subject Headings (MeSH)* and were appropriately modified for each database. All possible combinations of the following terms were used: dentin bonding, fiber post, post-luting, intra root canal, root canal dentin, root canal, ethanol application, ethanol wet-bonding, ethanol-saturated dentin, ethanol, pretreatment, wet-bonding, cementation techniques, dentin-bonding agent, luting cement, resin cement, self-adhesive resin cement, dual-cure composite luting cement, adhesion, bonding durability, bond durability, bond strength, dental bonding, durability, bonding longevity, bond longevity, longevity, push-out bond strength, and resin-dentin bond.

The following inclusion criteria were used for selecting the studies: (1) laboratory studies and (2) studies that compared ethanol wet-bonding and water wet-bonding. There was no restriction in the type of adhesive luting, resin cement or time of storage. Exclusion criteria were descriptive articles that did not compare pretreatment, review articles, case reports, opinion articles and academic theses.

Data collection and quality assessment

The eligibility of studies was determined by reading the titles and abstracts of articles identified in each database. The articles that met all inclusion criteria were selected and reviewed. The data retrieved were imported by EndNote X5 software (Thomson Reuters, EUA) and analyzed by two researchers (M.F.F.C, F.I.R.L; C.C.S.R). For the calibration exercise, the researchers discussed the criteria and applied them to a sample of 10% of the recovered studies. Calibration was performed to obtain excellent inter-examiner agreement (Kappa = 0.85). The information obtained was compared by reaching a consensus among examiners at the end of reading.

The analysis of the methodological consistency of the article as shown in Table 1 was performed according to a scale adapted as a modification of the Checklist/Scale adapted from.¹⁶ Disagreements were resolved by discussion and consensus between reviewers.

Statistical analysis

For the meta-analyses, two distinct data analyses were carried out: ethanol wet-bonding for 1 minute vs water wet-bonding in a bond strength test after 24 hours and 12 months. The pooled effect estimates were obtained by comparing the means to measure the possibility of comparing pretreatment, considering the subgroups and type of adhesive systems, etch-and-rinse and self-etch. This classification followed the type of classification indicated for the studies. For studies that evaluated different root thirds, the values were

extracted, and a mean was calculated to obtain the single sample size mean and standard deviation values for ethanol wet-bonding and water wet-bonding. In the selected studies, only the data of interest were extracted to be analyzed in the meta-analysis. If the study analyzed more than one type of adhesive system for the experimental and control group, the adhesive system chosen was the three-step etch-and-rinse system. If the study did not have a control group (water wet-bonding), the study was excluded from the analysis.

The significant differences between groups were investigated in Review Manager (RevMan version 5.3 software, Cochrane Collaboration, Copenhagen, Denmark, 2014) using a random-effects model. Significance was defined as $P \leq 0.05$ (Z test). The number of specimens was considered the number of experimental units. Forest plots were created to visualize the meta-analysis. Heterogeneity between studies was assessed by Cochran's Q test with a significance level of 0.10. The I^2 statistic was also examined, considering $I^2 > 60\%$ to indicate significant heterogeneity.

Results

Search and selection

The search results are shown in the flow diagram in Figure 1. The online search retrieved 456 references from PubMed, 222 from Web of Science, 163 from Scopus, 1 from BBO and 429 from Ovid. After the duplicate references were removed (523), the title and/or abstract of a total of 748 studies were read. A total of 722 studies were excluded based on the inclusion and exclusion criteria, and 26 were selected for full-text reading. Among these selected studies, 11 were excluded for the following reasons: studies reporting only coronal dentin,^{17,50} no comparison between groups,³¹ use of ethanol for irrigation,^{1,12,15,19,43,49} article not found³² and theses.⁴⁶ Fifteen studies were included in the review, and 7 articles were selected for the systematic review.^{2,6,8,10,11,13,47} However, studies were not included in the meta-analyses because of the type of adhesive system and/or storage time^{3,4,14} and time of application of ethanol.^{1,9,18,35,42,52}

Descriptive analysis

Table 2 shows the characteristics of the studies that compared pretreatment with ethanol wet-bonding and water wet-bonding in laboratory research. The studies were evaluated and described in ascending order of year of publication. The methodological characteristics of the studies are shown in Table 3. All studies were published between 2009 and 2017. The majority of the studies were conducted after 24 hours with pretreatment of ethanol for 1 minute,^{2,6,8,10,13,14,47} and push-out was the most common methodological test used. Moreover, 2 studies evaluated the groups using the microtensile bond-strength test.^{35,42} In this collection, 10 adhesive systems were evaluated including every commercially available adhesive classification: 3- and 2-step etch-and-rinse systems^{3,6,8,9,10,18,35,42} and 2- and 1-step self-etch systems.^{2,4,11,47} Most evaluated systems were 1) experimental resin-based systems in accordance with ethanol wet-bonding, considered in five studies,^{9,14,35,42,52} or 2) Scotchbond Multi-Purpose (3M ESPE), considered in four studies.^{6,10,13,35}

Meta-analyses

The meta-analyses were performed considering the type of pretreatment (ethanol wet-bonding and water wet-bonding) and storage time of the test (24 hours and 12 months) and considering the adhesive system as subgroup analyses (etch-and-rinse and self-etch systems). From the 7 studies included in the meta-analyses, 5 evaluated pretreatments with an etch-and-rinse system,^{6,8,10,13,47} and two evaluated pretreatments with a self-etching system for subgroup analysis.^{2,11}

The results of meta-analyses of pretreatment using ethanol wet-bonding are presented in Figures 2 and 3. In the global analysis in Figure 2, there was no difference between groups, showing no evidence that pretreatment with ethanol wet-bonding and water wet-bonding after 24 hours produces different bond strengths ($p=0.59$), and there was low heterogeneity ($I^2=0\%$). For the subgroup analyses, there were no differences between groups, showing no evidence that different types of adhesive systems produce different bond

strengths after 24 hours ($p=0.44$ and $p=0.98$). Low heterogeneity was observed for the etch-and-rinse adhesive system ($I^2=27\%$) and self-etching adhesive system ($I^2=0\%$). The analysis between pretreatment with ethanol wet-bonding and water wet-bonding with the etch-and-rinse adhesive system after 12 months (Figure 3) showed a significant difference between groups with low heterogeneity ($p=0.04$; $I^2=0\%$).

DISCUSSION

This systematic review summarizes the *in vitro* data on the influence of ethanol wet-bonding on the retention of fiber posts in root canals. Different adhesive systems—etch&rise, self-etch, experimental adhesive—and luting cements—dual-cure and self-adhesive resin cement—have been tested in the literature in an attempt to increase the retention of fiber posts in root canals. The hypothesis that the application of ethanol wet-bonding does not improve the durability of the adhesion of fiber posts on root dentin was accepted.

A significant problem in adhesive restorations was the degradation of the hybrid layer.¹¹ For this purpose, different pretreatments of root dentin have been tested to improve the immediate and long-term bond strength between fiber posts and root dentin.^{2,310,11,13} Pretreatment procedures aim to facilitate interactions between different substrates and consequently optimize adhesion.²⁹ The present study indicated that ethanol wet-bonding does not improve the retention of fiber posts when compared with water wet-bonding after 24 hours. Water wet-bonding has been recommended for dentin bonding for years⁴² to prevent problems with collagen collapse after acid-etching that results in resin infiltration.^{25,42} This technique can expand the dentin matrix because the greater hydrogen bonding forces break interpeptide H-bonds.^{30,33} However, when the spaces are filled with water, solvated comonomers present in the adhesives may not be able to remove and replace all the water.⁴⁰ At the same time, not all adhesive monomers are soluble in water, and the production of phase changes in water-saturated dentin is a possibility.³⁴ Pretreatment with ethanol wet-bonding is based on the concept that ethanol is responsible for replacing water from dentin and dissolving monomers.^{8,37} This technique could

contribute to making the collagen matrix more hydrophobic and less micropermeable.⁴ However, a reasonable explanation for the result was that the simplified technique using ethanol for 1 minute was extremely technique-sensitive and does not completely replace the water in fibrillary spaces.⁹

At the end of 12 months, when longevity was evaluated, pretreatment with ethanol for etch-and-rise adhesive systems does not preserve the bond strength of the fiber glass to the root dentin. Despite the fact that the result for pooled analyses was not a significant result to ethanol wet-bonding, the characteristics presented in the technique indicated an advantage when hydrophobic adhesives were used.^{9,14} This characteristic can be explained by the fact that ethanol was less capable of breaking spontaneously and forming hydrogen bonds with collagen fibrils, and it increased the ability of ethanol-saturated collagen fibrils to form interpeptide bonds on the collagen matrix.³⁷ This process prevents interpeptide H-bonding, and interfibrillar spaces serve as diffusion channels for monomers.³⁴ As a consequence, the interfibrillar spaces of ethanol-saturated dentin are larger than those of water wet-bonding.^{23,52} Indeed, more adhesive monomers could penetrate into ethanol-saturated dentin^{34,35} and reduce the plasticizing effect of water.⁵⁰

Clinicians have increased the use of fiber posts.^{39,45} Nevertheless, the main reason for failure is still debonding with a decrease in retention at the dentin/resin interface.³⁶ This failure might be a result of intrinsic factors and hydrolytic degradation.^{10,11} The findings of this study indicate that when the posts were luted with conventional pretreatment into root canals, the combination of the etch-and-rinse system + dual-cure resin cement significantly improved the durability of post retention. A reasonable explanation for the ethanol not being able to improve bonding as water wet-bonding could be related to the short time application. For this, ethanol could rapidly evaporate or be replaced by water permeating from open and funneled dentin tubules after smear-layer removal.⁴¹ Furthermore, ethanol treatment also shrinks the water-trapping interfibrillar glycosaminoglycans.²⁷

Most studies included in this review performed the ethanol wet-bonding technique with an experimental adhesive, hydrophobic compositions, or a

commercial adhesive system, Scotchbond Multi-Purpose (3M ESPE), with less hydrophilic behavior. Thus, the use of pretreatment associated with hydrophobic monomers, even relatively hydrophobic resins,⁷ produces better infiltrate in the ethanol-saturated demineralized dentin into the matrix,⁹ thereby creating greater stiffness and stability at the adhesive layer than hydrophilic adhesives.⁴² Moreover, the longevity of resin-dentin bonds is directly related to the hydrophobicity of the resin adhesives.²⁶ The hydrophobic resin could also help achieve stronger and more durable dentin bonds,¹⁴ since they have less water sorption, solubility and rate of water diffusion.²⁶

Different mechanical tests to measure bond strength are reported in the *in vitro* literature, and among the mechanical tests used in the included studies, push-out was the test most often used. A total of 86.6% of studies evaluated the bond strength of fiber posts in root canals and compared the effect of ethanol wet-bonding using this test. The main aspect of the push-out test that differentiates it from tensile and shear tests in flat, bonded interfaces is the confinement of the composite within the walls of the prepared cavity, more akin to the conditions found in the clinic.⁵ Furthermore, the possibilities of premature failure are reduced when compared to the trimming and non-trimming microtensile tests.²⁰

All of included *in vitro* studies had a moderate risk of bias, demonstrating that variables like information on sample size calculation and who implemented the random allocation and blinding of the operator could influence the results. However, the risk of bias assessment can be subjective and should be interpreted as such.²⁹ Despite the moderate risk, the heterogeneity among the studies was low ($I^2=27\%$ and $I^2=0\%$).

This meta-analysis identified positive evidence for water wet-bonding technique after storage. At the same time, this pooled analysis was performed with *in vitro* studies, and only 2 studies were possible to analyzed after 12 months. For this reason, the results of the systematic review should be interpreted with caution, as *in vitro* studies have limitations from simulating *in vivo* conditions.²⁹ It is essential to conduct longitudinal studies with representative samples to determine whether use of ethanol wet-bonding could

result in improved clinical success rates for fiber post restorations. Therefore, the overall results of the present study favor pretreatment for the durability and retention of fiber posts.

CONCLUSION

The analysis of the *in vitro* literature indicates that simplified ethanol wet-bonding does not improve the retention of fiber posts in root canals immediately or after 12 months.

Clinical Relevance: For the necessity to improvement the bond strength to root canal, ethanol wet-bonding can be an alternative. However, *in vivo* studies are needed to assess for long-term effects.

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Table 1. Description of criteria for evaluating the quality of the selected articles.

Criteria	Classification	Score	Definition
1. Intervention	Adequate	1.0	The intervention for each group, including how and when it was administered, with sufficient detail to enable replication
	Inadequate	0.5	Data not clearly presented in the article
	Not described	0.0	Insufficient data
2. Outcomes	Adequate	1.0	Completely defined, pre-specified primary and secondary outcome measures, including how and when they were assessed
	Inadequate	0.5	Insufficient data
	Not described	0.0	Data not clearly presented in the article
3. Sample size	Adequate	1.0	How sample size was determined
	Not described	0.0	Does not describe/present/communicate the way sample calculation was done
4. Randomization	Adequate	1.0	Method used to generate and to implement the random allocation sequence, describing the steps taken to conceal the sequence until intervention was assigned
	Inadequate	0.5	Insufficient data
	Not described	0.0	Data not clearly presented in the article
5. Implementation	Adequate	1.0	Who generated the random allocation sequence, who enrolled patients' (teeth), and who assigned teeth to intervention
	Inadequate	0.5	Insufficient data
	Not described	0.0	Data not clearly presented in the article
6. Blinding	Adequate	1.0	If done, who was blinded after assignment to intervention and how
	Inadequate	0.5	Insufficient data
	Not described	0.0	Data not clearly presented in the article
7. Statistical methods	Adequate	1.0	Statistical treatment fully described and adequate
	Inadequate	0.5	Statistical treatment not fully described and inadequate
	Not described	0.0	No statistical treatment applied

Checklist/Scale adapted for this study from Faggion Jr (2012)

Table 2. Description data from included studies according to substrate, pre-treatment, type of test, storage and conclusion.

Author,Year	Substrate	Sample Size per group	Pre-treatment	Adhesive System/Resin cement	Bond Strength Test	Storage	Conclusion
Carvalho 2009	Single-canal premolars	5	99.6% ethanol-wet bonding application for 1 minute versus water wet bonding technique	etch-and-rinse adhesive system and dual-cure composite luting cement	Push-out	24 hours	Ethanol-wet bonding technique in intraradicular dentine can improve the bond strength compared with traditional water-wet bonding technique, when a three-step ethanol-based bonding system is used
Cecchin 2011a	Bovine incisors	10	100% ethanol-wet bonding application for 1 minute versus no treatment	etch-and-rinse adhesive system and dual-cure composite luting cement	Push-out	24 hours and 12 months	The use of ethanol-wet bonding did not preserve the bond strength of the adhesive system
Cecchin 2011b	Bovine incisors	15	100% ethanol-wet bonding application for 1 minute versus no treatment	self-etching adhesive system and dual-cure composite luting cement	Push-out	24 hours and 12 months	The use of CHX and/or EtOH pretreatment produced no effect on the immediate groups and was capable of preserving the bond strength of the fiber post relined with resin composite to root dentin for 12 months.
Sauro 2011	Upper central human incisors	10	100% ethanol-wet bonding application for 3 minutes versus water wet dentin	experimental resin and etch-and-rinse adhesive system and flowable resin composite	Microtensile	24 hours	Ethanol-wet bonding technique significantly increased the bond strength of the tested resins to root canal dentin.
Carvalho 2012	Single-rooted premolars	5	99.6% ethanol-wet bonding application for 30 s versus water wet bonding technique	experimental resin and etch-and-rinse adhesive system and unfilled resin	Push-out	24 hours	Simplified ethanol-wet bonding technique is not sufficient to enhance the push-out strength in the root canal using the tested materials
Pei 2012	Single-rooted premolars	15	100% ethanol-wet bonding application for 20 s, 60 s or three 60 s periods and stepwise application versus no treatment	experimental resin and etch-and-rinse adhesive system and resin composite	Microtensile	24 hours	Root dentine treated with stepwise ethanol application significantly improved the bond effectiveness of hydrophobic adhesive, whereas ethanol treatment for less than one minute should be avoided.
Alaghemand 2013	Upper central human incisors and canine	12	99.6% ethanol-wet bonding application for 1 minute versus water wet bonding technique	self-etching adhesive system and dual-cure composite luting cement	Push-out	24 hours	The application of different pretreatment solutions did not improve the bond strength of the adhesive system used to cement fiber post to root dentine
Bitter 2014	Central incisors	20	99% ethanol-wet bonding application for 1 minute versus water wet bonding technique	etch-and-rinse adhesive system and dual-cure	Push-out	7 days and thermocycling	Pretreatment of the root canal using ethanol significantly increased bond

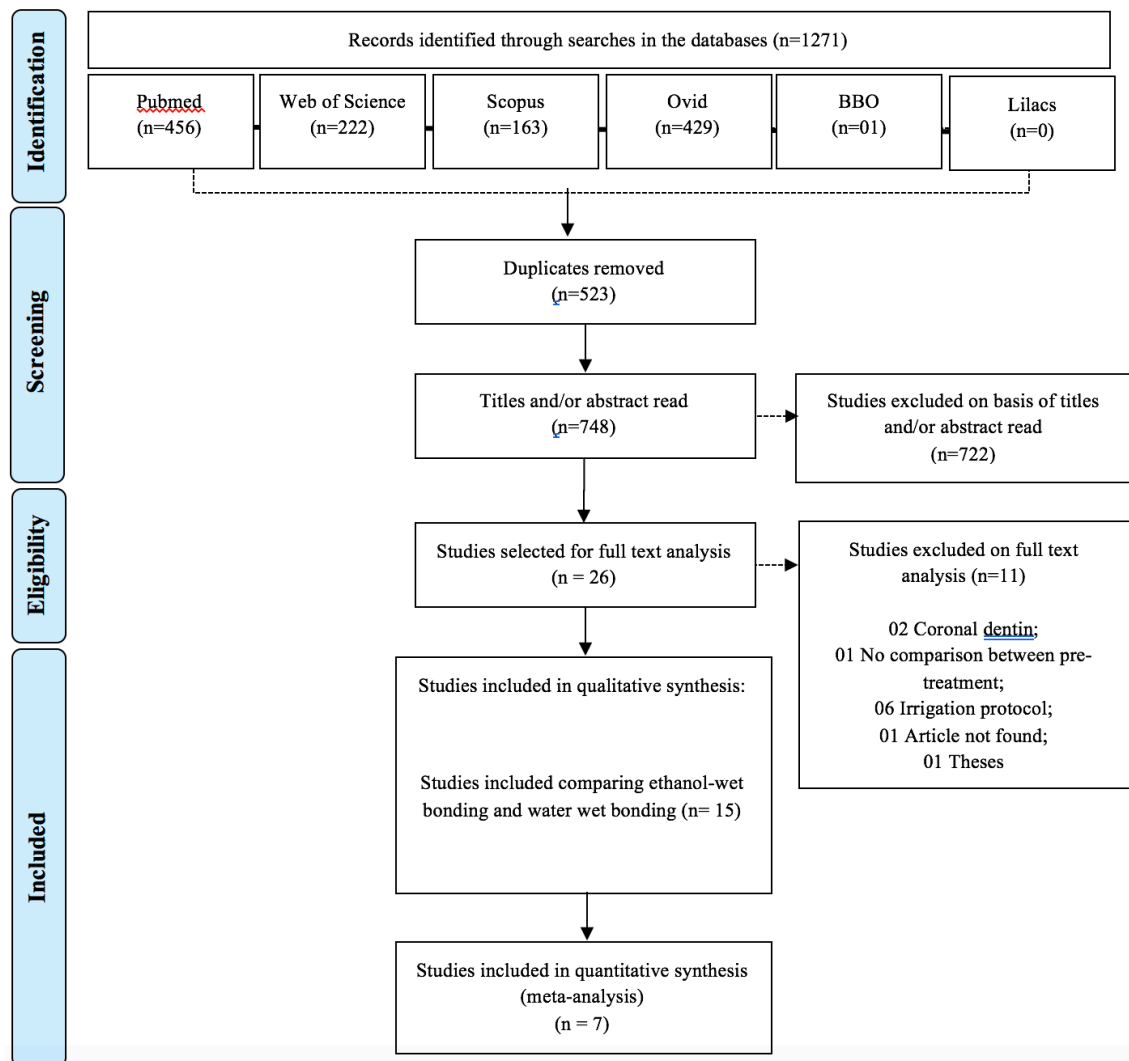
				composite luting cement and self-adhesive resin cement			(10,000 cycles, 5–55 °C)/stored for 6 months	strength of fiber posts luted with an etch-and-rinse adhesive system and a self-adhesive resin cement.
Bordin 2014	Bovine incisors	10	100% ethanol-wet bonding application for 1 minute versus no treatment	etch-and-rinse adhesive system and dual-cure bonding agent	Push-out	24 hours and 12 months	Ethanol application, prior to the use of an adhesive system which solvent is a mixture of water and ethanol, helps to maintain the long-term bond strength of fiber-glass posts cemented to the root dentine	
Cecchin 2014	Bovine incisors	10	100% ethanol-wet bonding application for 1 minute versus no treatment	etch-and-rinse adhesive system and dual-cure composite luting cement	Push-out	Thermocycling (250,000 cycles, 37 °C)	Ethanol pretreatment preserved the bond strength of the fiber post after cyclic loading when the etch-and-rinse adhesive system Adper Scotchbond Multi-Purpose was used	
Ekabaram 2014	Single-rooted	24	100% ethanol-wet bonding application for 1 minute versus no treatment	experimental resin and dual-cure composite luting cement	Push-out	24 hours, 6 and 12 months	The addition of 2% chlorhexidine to ethanol-wet bonding with a hydrophobic etch-and-rinse adhesive did not further improve the bond strength of luting of a fiber post to intrarradicular dentine, when compared to ethanol-wet bonding alone after 12 months of ageing	
Zavattini 2014	Central incisors	20	100% ethanol-wet bonding application for 5 minute (seven successive applications) versus water wet bonding technique	experimental resin and dual-cure	Push-out	24 hours	Ethanol wet-bonding improves the adhesion of common resin-based materials (no reactive fillers) in deeper root canal areas	
França 2015	Bovine incisors	18	100% ethanol-wet bonding application for 1 minute versus water wet bonding technique	etch-and-rinse adhesive system and dual-cure composite luting cement	Push-out	48 hours and 180 days	Ethanol improved push-out bond strength to intrarradicular dentin, with the former providing the best results, regardless of the storage time	
Victorino 2016	Canines	10	99% ethanol-wet bonding application for 1 minute versus water wet bonding technique	self-etching adhesive system and dual-cure composite luting cement	Push-out	24 hours	Root dentin pretreatment with ethanol increased the bond strength values of fiber post cemented with resin cement, in comparison to the dentin pretreatment	

									with distilled water
Bitter 2017	Central incisors	10	100% ethanol-wet bonding application for 1 minute versus water wet bonding technique	self-etching adhesive system and dual-cure composite luting cement and self-adhesive resin cement	Push-out	24 hours, thermocycling (10,000 cycles, 5-55 °C)/stored for 6 months	Ethanol had no positive effects on bond strength inside the root canal, whereas self-etch adhesive strategies benefit from ethanol application in the apical part of the post space preparation		

Table 3. Quality assessment of the selected studies, research quality or methodological soundness: high (≥ 5), moderate (3 - 5) or low (< 3).

Article	Intervention	Outcomes	Sample size	Randomization	Implementation	Blinding	Statistical methods	Total points	Research quality
Carvalho 2009	1	1	0	0,5	0	0	1	3,5	Moderate
Cecchin 2011a	1	1	0	0,5	0	0	1	3,5	Moderate
Cecchin 2011b	1	1	0	0,5	0	0	1	3,5	Moderate
Sauro 2011	1	1	0	0	0	0	1	3	Moderate
Carvalho 2012	1	1	1	0,5	0	0	1	4,5	Moderate
Pei 2012	1	1	0	0,5	0	0	1	3,5	Moderate
Alaghemand 2013	1	1	0	0,5	0	0	1	3,5	Moderate
Bitter 2014	1	1	0	0,5	0	0	1	3,5	Moderate
Bordin 2014	1	1	1	0,5	0	0	1	4,5	Moderate
Cecchin 2014	1	1	0	0	0	0	1	3	Moderate
Ekabaram 2014	1	1	0	0,5	0	0	1	3,5	Moderate
Zavattini 2014	1	1	0	0,5	0	0	1	3,5	Moderate
França 2015	1	1	0	0,5	0	0	1	3,5	Moderate
Victorino 2016	1	0,5	0	0,5	0	0	1	3	Moderate
Bitter 2017	1	1	0	0,5	0	0	1	3,5	Moderate

Figure 1. Four-phase PRISMA flow-diagram for study collection, showing the number of studies identified, screened, eligible, and included in the review and meta-analysis.



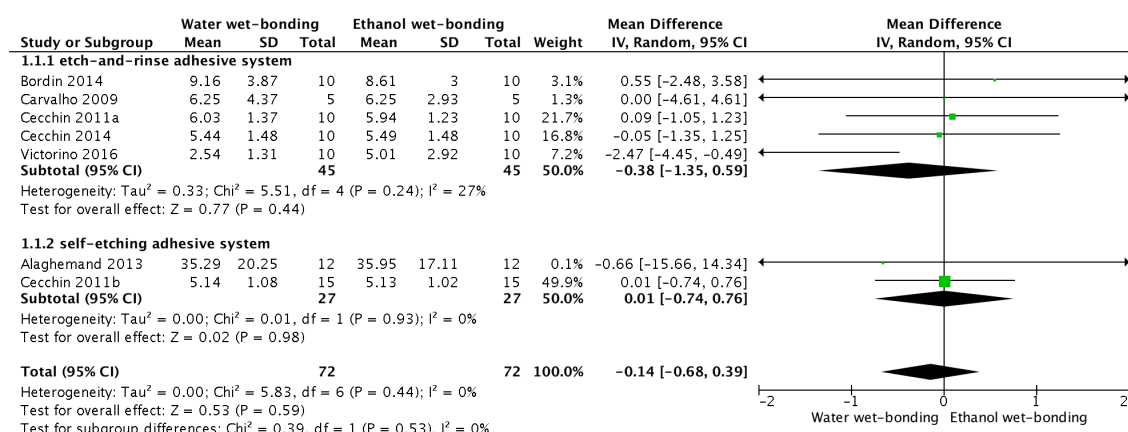


Figure 2. Forest plot for the analysis of shear bond strength after 24 hours between ethanol-wet bonding and water wet bonding

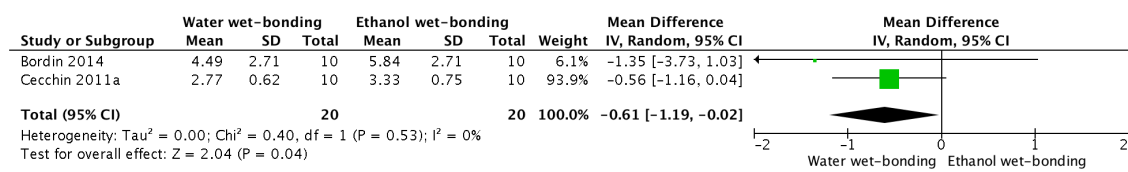


Figure 3. Forest plot for the analysis of shear bond strength after 12 months with etch-and-rinse adhesive system between ethanol-wet bonding and water wet bonding

Trabalhos publicados em anais de congresso

Carvalho MFF, Bicalho AA, Soares CJ, Santa-Rosa CC, Magalhães CS, Yamauti M, Moreira AN. Influência da técnica alcóolica simplificada na cimentação de pinos intrarradiculares. In: SBPQO, 2017, Campinas. SBPqO - Sociedade Brasileira de Pesquisa Odontológica, 2017, v. 31, p. 253.

Limeira FIR, Santos CC, Nascimento VV, **Carvalho MFF**, Leite CF, Yamauti M, Moreira AN, Magalhães CS. Influência da idade na composição química das dentinas radiculares humanas e bovinas nos diferentes terços radiculares. In: SBPQO, 2017, Campinas. SBPqO - Sociedade Brasileira de Pesquisa Odontológica, 2017, v. 31, p. 301.

Sá TCM, **Carvalho MFF**, Limeira FIR, Santa-Rosa CC, Magalhães CS, Yamauti M, Moreira AN. Resistência de união na fixação de pinos intrarradiculares de acordo com diferentes pré-tratamentos dentinários. . In: SBPQO, 2017, Campinas. SBPqO - Sociedade Brasileira de Pesquisa Odontológica, 2017, v. 31, p.304.

Monize Ferreira Figueiredo de CARVALHO, Amanda Carolina Neija LEIJÔTO, Francisco Iverson Rodrigues LIMEIRA, Caroline Chistine SANTA-ROSA; Monica YAMAUTI; Allyson Nogueira MOREIRA. Resistência de união de um cimento resinoso autoadesivo empregados na fixação de pinos intrarradiculares pré-fabricados de acordo com diferentes pré-tratamentos dentinários. In: XIII Encontro Científico da Faculdade de Odontologia, 2016, v.52, p. 26.

Alef Gimenes ARAÚJO, **Monize Ferreira Figueiredo de CARVALHO**, Caroline Chistine SANTA-ROSA, Lais Ferreira SANTOS, Monica YAMAUTI; Allyson Nogueira MOREIRA. Influência da técnica alcóolica simplificada na resistência de união de um cimento resinoso. In: XIII Encontro Científico da Faculdade de Odontologia, 2016, v.52, p. 6.

Leijôto ACN, Bicalho AA, Soares CJ, Almeida, GC, **Carvalho MFF**, Moreira NA, Yamauti M. Avaliação da interface de cimentação de pinos intrarradiculares de acordo com diferentes técnicas de preparo de conduto radicular. In: GBMD, 2016, Uberlândia, 2016.

Carvalho MFF, Santos LCS, Watanabe E, Tavano KTA, Botelho AM, Moreira NA. Avaliação do fluoreto de sódio a 2% como um novo método para desinfecção de dentes humanos extraídos. In: SBPQO, 2015, Águas de Lindóia. Campinas. SBPqO - Sociedade Brasileira de Pesquisa Odontológica, 2015. v.29. p.292.

De Couto AM, **Carvalho MFF**, Fernandes IB, Dourado MR, Mesquita ATM, Ramos-Jorge ML. Câncer de cabeça e pescoço entre usuários de maconha: uma meta-análise de estudos de caso-controle. In: SBPQO, 2014, Águas de Lindóia. Proceedings of the 31st 8 SBPqO Annual Meeting), 2014. v.28.

Atividades de ensino

Professora voluntária do "Curso de Auxiliar de Saúde Bucal da UFMG". 32 horas-aula semestral (2015-2016).

Ministrou aula intitulada "Doença Periodontal e Decisão de tratamento em idosos" na disciplina de Tópicos em Estética Restauradora/Saúde do Idoso da UFMG, 26 de setembro de 2016.

Professora substituta do Departamento de Odontologia Restauradora da UFMG. 20 horas-aula no período. (09/2017 a 11/2017).

Banca

Defesa de conclusão de curso: "Avaliação da influência da técnica alcoólica simplificada na resistência de união com uso de um cimento resinoso autoadesivo, UFMG, 27 de novembro de 2017.

Defesa de conclusão de curso: "Avaliação da influência da técnica alcoólica simplificada na resistência de união com utilizando um cimento resinosos dual, UFMG, 27 de novembro de 2017.

Participação em eventos

31º Reunião Anual da Sociedade Brasileira de Pesquisa. Câncer de cabeça e pescoço entre usuários de maconha: uma meta-análise de estudos de caso-controle. 2014. (Congresso).

Participação como ouvinte: “Importância da interface adesiva na longevidade de restaurações de resina compostas”, 2015. 4 horas-aula.

32º Reunião Anual da Sociedade Brasileira de Pesquisa. Avaliação do fluoreto de sódio a 2% como um novo método para desinfecção de dentes humanos extraídos. 2015. (Congresso).

Treinamento operacional: Máquina Universal de Ensaio de longo curso. 25 a 29 de abril, 2016.

52º Encontro do Grupo Brasileiro de Materiais Dentários. Avaliação da interface de cimentação de pinos intrarradiculares de acordo com diferentes técnicas de preparo de conduto radicular. 2016. (Congresso).

Participação como comissão avaliadora de trabalhos científicos no XIII Encontro Científico da Faculdade de Odontologia da Universidade de Minas Gerais. 2016. (Encontro).

Participação como ouvinte no XIII Encontro Científico da Faculdade de Odontologia da Universidade de Minas Gerais. 2016. (Encontro).

Participação como comissão avaliadora de trabalhos científicos do 13º Congresso Internacional de Odontologia de Minas Gerais. 2016. 24 horas-aula. (Congresso).

Participação como ouvinte na 69ª Reunião Anual da Sociedade Brasileira para o Progresso da Ciência. 2017. (Reunião).

34º Reunião Anual da Sociedade Brasileira de Pesquisa. Resistência de união na fixação de pinos intrarradiculares de acordo com diferentes pré-tratamentos dentinários. 2017. (Congresso).