



UNESP - Universidade Estadual Paulista
“Júlio de Mesquita Filho”
Faculdade de Odontologia de Araraquara



Aline Carvalho Giroto

**Avaliação da adaptação marginal interna, resistência de união e resistência à
fratura da associação entre resinas bulk-fill e adesivos simplificados em
dentes tratados endodonticamente**

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A Deus, pelas pessoas e caminhos que colocou em minha vida. Por todos os sonhos, vontade e dedicação para alcança-los.

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Giroto A. Avaliação da adaptação interna, resistência de união e resistência à fratura da associação entre resinas bulk-fill e adesivos simplificados em dentes tratados endodonticamente [tese de doutorado]. Araraquara: Faculdade de Odontologia da UNESP; 2019.

RESUMO

O objetivo do presente estudo foi avaliar a adaptação interna (AI), resistência de união (RU) e resistência à fratura (RF) da associação entre resinas bulk-fill de alta viscosidade e adesivos simplificados, quando utilizados para restaurar dentes que foram endodonticamente tratados. Foram avaliados 4 sistemas restauradores comerciais, sendo eles Grupo Controle (GC): Scotchbond Multi-Purpose + Filtek Z350XT (3M/ESPE); (SBU/FBF) Single Bond Universal + Filtek Bulk-fill (3M/ESPE); (OBA/SF) OptBond All-in-one + Sonic Fill (Kerr) e (TBU/TBF) Tetric-N- Bond Universal + Tetric-N-Ceram Bulk-Fill (Ivoclar/Vivadent). Em 32 pré-molares humanos, a superfície oclusal foi planificada, cavidades classe I padronizadas foram confeccionadas e os dentes foram tratados endodonticamente. A irrigação foi feita com hipoclorito de sódio (NaOCl) 2,5% e EDTA 17%. Após a obturação (AH Plus, Dentsply, De Trey, Konstanz, Germany) e a limpeza da câmara pulpar com álcool 99%, foi aplicada uma fina camada de cimento de ionômero de vidro modificado por resina Ionoseal (VOCO, GmbH, Vuxhaven, Alemanha). Os dentes foram então divididos nos grupos experimentais (n=8), e as restaurações foram realizadas. Após 24 horas, os espécimes receberam um corte centralizado, as metades obtidas foram polidas e réplicas em resina epóxi para a avaliação da AI em microscopia eletrônica de varredura foram confeccionadas. Em 40 terceiros molares humanos, um corte foi feito para exposição da dentina profunda. Após a padronização da smear layer (lixa #600), este tecido foi tratado com NaOCl 2,5% por 30 min, EDTA 17% por 3 min, impregnação com cimento AH Plus o qual ficou em contato com a dentina por 5min e limpeza com álcool 99%. Os espécimes foram divididos aleatoriamente nos 4 grupos experimentais (n=10), após a aplicação do sistema adesivo um bloco com a respectiva resina foi confeccionado. Após 24 horas os espécimes foram seccionados e submetidos ao teste de microtração. O ensaio foi realizado em dois tempos: após 24 horas e após 1 ano de armazenamento em água destilada. Em 50 pré-molares humanos, o tratamento endodôntico foi realizado conforme descrito anteriormente. Neste estudo além de serem avaliados os grupos experimentais também foi avaliado um controle negativo (GCN) (n=10) constituído de dentes tratados endodonticamente sem restauração. O teste de RF foi realizado na máquina de ensaios universal. Os dados foram avaliados através do teste de ANOVA e, quando necessário ao teste de Tukey, com nível de significância de 5%. Quanto à AI, a maior porcentagem de GAPs formou-se para a resina SonicFill e não houve diferença estatística entre as demais resinas estudadas ($p>0,05$). No período de 24 horas, não houve diferença estatisticamente significativa na RU entre os sistemas adesivos estudados ($p>0,05$). No entanto, após o armazenamento de 1 ano, o SBU foi o único que não apresentou redução da RU ($p<0,05$), não houve diferença estatisticamente significativa dos resultados encontrados após 24 horas ($p>0,05$). Os adesivos simplificados OBA e SBU apresentaram os mesmos valores de RU que o sistema SBMP (Condiciona e lava de 3 passos) ($p>0,05$). O TBU além de apresentar uma redução da RU após um ano, também teve os piores resultados de RU ($p<0,05$). Quanto à avaliação da RF o grupo controle negativo (GCN, 215,14 N) apresentou os menores valores de resistência ($p<0,05$). Não foram encontradas diferenças estatisticamente significantes

entre os demais grupos que estavam restaurados ($p>0,05$). Conclui-se que a associação entre resinas bulk-fill e adesivos simplificados, comparado aos sistemas convencionais não apresentaram diferenças quanto a resistência a fratura. Quanto a adaptação interna o sistema OBA/SF apresentou os piores resultados. Por último, inicialmente não houve diferença entre os adesivos quanto a RU. Após 1 ano os adesivos simplificados OBA e SBU tiveram os mesmos valores de RU que o sistema SBMP (Condiciona e lava de 3 passos). SBU não apresentou redução da RU. TBU apresentou redução da RU e os piores resultados, comparado aos demais sistemas adesivos.

Palavras chave: Adesivos dentinários. Endodontia. Restauração dentária permanente. Dentina.

Giroto A. Evaluation of internal adaptation, bond strength, and fracture resistance of the combination of high-viscosity bulk-fill resins and simplified adhesives in the restoration of endodontically-treated teeth [tese de doutorado]. Araraquara: Faculdade de Odontologia da UNESP; 2019.

ABSTRACT

The objective of this study was to evaluate internal adaptation (IA), bond strength (BS), and fracture resistance (FR) of the combination of high-viscosity bulk-fill resins and simplified adhesives in the restoration of endodontically-treated teeth. Four commercial restoration systems were evaluated, which were the control group (CG) in which Scotchbond Multi-Purpose + Filtek Z350XT (3M/ESPE) was used, (SBU/GBF) Single Bond Universal + Filtek Bulk-fill (3M/ESPE); (OBA/SF) OptBond All-in-one + Sonic Fill (Kerr), and (TBU/TBF) Tetric-N- Bond Universal + Tetric-N-Ceram Bulk-Fill (Ivoclar/Vivadent). In 32 human pre-molars, the occlusal surface was flattened, standard class I cavities were created, and the teeth were treated endodontically. Irrigation was performed using 2.5% sodium hypochlorite (NaOCl) and 17% EDTA. After the fillings (AH Plus, Dentsply, De Trey, Konstanz, Germany) and cleaning of the pulp chamber with 99% ethanol, a thin layer of ionoseal resin-modified glass ionomer luting cement was applied (Voco, GmbH, Vuxhaven, Germany). The teeth were then divided into the experimental groups (n=8) and the restorations were performed. After 24 hours, the specimens were sectioned in the middle and the halves obtained were polished, and replicas were made in epoxy resin to evaluate IA using scanning electron microscopy. In 40 human molar thirds, a cut was made to expose the deep dentin. After standardization of the smear layer (file # 600), the tissue was treated with 2.5% NaOCl for 30 min and 17% EDTA for 3 min. It was then impregnated with AH Plus cement, which was in contact with the dentin for 5 min. Finally, the samples were cleaned with 99% ethanol. The specimens were randomly divided into the four experimental groups (n=10). After the application of the adhesive system, a block was made with the respective resin. After 24 hours the specimens were sectioned, and the microtension test was applied. The assay was performed in two stages: after 24 hours and after 1 year of storage in distilled water. In 50 human premolars, endodontic treatment was performed as previously described. In addition to the experimental groups, a negative control (n=10) consisting of endodontically treated teeth without restoration was also evaluated. The FR test was performed using the universal testing machine. The data were evaluated using the ANOVA test and, if necessary, Tukey's test, with a significance level of 5%. In the case of AI, the highest percentage of GAPs was formed for the SonicFill resin, and there were no statistical differences between the other resins studied ($p>0.05$). After 24 hours, there were no statistically significant differences in μ TBS between the adhesive systems studied ($p>0.05$). However, after 1 year of storage, just the Single Bond Universal did not presented reduction in μ TBS and were not significant different from the results obtained after 24 hours ($p<0.05$). The simplified adhesives OBA and SBU showed similar μ TBS values than the 3 step etch-and-rinse adhesive system SBMP ($p>0,05$). As for the FR assessment, the negative control group (NCG, 215.14 N) exhibited the lowest resistance values ($p<0.05$). No statistically significant differences were found between the other groups that were restored ($p>0.05$). It was concluded that the association between bulk-fill resins and universal adhesives did not differ in fracture resistance when compared to conventional systems. When internal adaptation was considered, the OBA/SF system exhibited the worst results. Finally, after 24 hours there were no difference in μ TBS values between

the adhesives systems studied. After one year the simplified adhesives OBA and SBU showed similar μ TBS values than the 3 step etch-and-rinse adhesive system SBMP. SBU did not presented μ TBS reduction and TBU presented the worst results between the adhesive systems studied.

Keywords: Dentin-bonding agents. Endodontics. Dental restoration, permanent. Dentin.

SUMÁRIO

1 INTRODUÇÃO	9
2 PROPOSIÇÃO	12
3 PUBLICAÇÃO.....	13
3.1 Publicação 1	13
3.2 Publicação 2	29
3.3 Publicação 3	45
4 DISCUSSÃO	56
5 CONCLUSÃO	64
REFERÊNCIAS	65
APÊNDICE	71
ANEXO	77

1 INTRODUÇÃO

É fundamental um adequado selamento coronário para o sucesso da terapia endodôntica a longo prazo^{1,2,3}. Nos dentes tratados endodonticamente, o procedimento restaurador devolve estética, propriedades biomecânicas, além de prevenir a contaminação bacteriana e consequente reinfecção do canal radicular^{3,4}. Além disso, o alargamento dos canais associado a utilização de agentes químicos, como os irrigantes e os cimentos, compromete a integridade estrutural dos dentes que foram tratados^{5,6}. Como consequência, um bom selamento coronal torna-se imprescindível uma vez que a taxa de fratura e o risco de perda do dente é maior quando comparado aos dentes vitais⁷⁻⁹.

Restaurar dentes endodonticamente tratados é desafiador e exige tanto técnica como a utilização materiais restauradores adequados^{10,11}. Os materiais utilizados idealmente devem ser biocompatíveis ao meio bucal, resistentes às cargas oclusais e ao desgaste, duradouros, capazes de se aderir às estruturas dentais, apresentar estética aceitável, ser de fácil aplicação e possibilitar o preparo minimamente invasivo para permitir a preservação da estrutura dental remanescente¹², a qual geralmente encontra-se fragilizada⁵⁻⁷. Assim, dentre todos os materiais, os sistemas adesivos e as resinas compostas são os que melhor atendem essas exigências^{13,14}, especialmente para nos casos de moderada perda de estrutura coronária³.

Entretanto, um dos problemas das resinas compostas é a tensão de polimerização¹⁵, a qual pode ter como consequência uma série de complicações clínicas como trincas na estrutura dental, deflexão de cúspides, formação de fendas na interface entre o dente e a restauração, descolamento, manchamento marginal, cáries secundárias e microinfiltração¹⁶⁻¹⁸. Além disso, as resinas possuem uma profundidade de polimerização limitada¹⁹, como resultado, a utilização de grandes incrementos pode comprometer as propriedades mecânicas do material²⁰. Uma das alternativas que vem sendo utilizada para minimizar estes problemas é a técnica incremental, que consiste na aplicação da resina composta em incrementos horizontais ou oblíquos de até 2mm²¹, assim o fator de configuração cavitário é minimizado pela redução da área de contato entre a resina e as paredes do preparo cavitário, e acredita-se que estes procedimentos reduzem a contração de polimerização final do material e minimize a formação de GAPs internos²².

No entanto, a aplicação de múltiplos incrementos consome tempo e torna a técnica mais complexa²³, além de aumentar o risco de ficarem vazios no interior da

restauração²⁴. Com o objetivo de superar as deficiências da técnica incremental e diminuir o tempo clínico do procedimento restaurador vem sendo introduzido no mercado odontológico um novo conceito de material restaurador, que são as resinas compostas Bulk-fill. Os fabricantes desses novos compósitos afirmam que os mesmos podem ser eficientemente polimerizados em profundidades de até 4-5mm²⁵ e ao mesmo tempo manter uma baixa tensão de polimerização²⁶. Além das vantagens potenciais para simplificar a técnica e economizar tempo, a aplicação em incrementos maiores evita a incorporação de vazios e a contaminação entre as camadas, deixando assim as restaurações mais compactas²⁷. Quanto a apresentação comercial estes compósitos estão disponíveis em alta e baixa viscosidade. Os compósitos de baixa viscosidade, conhecidos como resinas bulk-fill flow, necessitam da aplicação de uma camada final de resina composta de cobertura, a qual deve ser feita com compósito convencional²⁸. Já os compósitos de alta viscosidade, também chamados de compósitos bulk-fill esculpíveis, são indicados para o uso sem recobrimento e podem ser aplicados como compósitos de passo único²⁹.

A fotopolimerização de incrementos com 4mm de espessura ou mais foi possível graças a mudanças feitas na formulação desses materiais. O aumento da translucidez desses compósitos permitiu uma ótima transmissão da luz³⁰. O alívio da tensão de polimerização ocorreu através uma modulação na reação de polimerização através da utilização de monômeros especiais. Também estão sendo utilizados fotoinicadores mais reativos, os quais aumentam significativamente a reatividade do monômero e a profundidade de polimerização³¹. E por último também foram adicionadas diferentes tipos de partículas de carga, as quais são capazes de absorver a tensão além de aumentar a resistência mecânica²⁷. Estudos laboratoriais vem provando a adequada profundidade de presa desse materiais³². Além disso outras propriedades mecânicas são semelhantes aos compósitos convencionais. A contração e a tensão de polimerização é igual ou mesmo menor do que as resinas compostas convencionais³³, além disso o *creep* desses materiais também pode ser considerado adequado³⁴.

Os sistemas adesivos possuem um papel fundamental no desempenho das restaurações confeccionadas com resinas compostas Bulk-fill³⁵. No estudo conduzido por Roggendorf et al.³⁵, 2011 os sistemas adesivos constituíram o fator que mais afetou a qualidade marginal de restauração feitas com esse material. Quando a tensão de polimerização excede a resistência de união, ocorre um descolamento do material

restaurador^{36,37}. Assim, a formação de GAPs e a microinfiltração podem ser causadas pelas falhas adesivas, e sabe-se que o prognóstico da terapia endodôntica pode ser influenciado pela contaminação radicular através de lacunas marginais ou internas da restauração coronal³⁸. E no conceito de simplificação, tem sido muito recomendado pelos fabricantes o uso de adesivos simplificados juntamente com os compósitos Bulk-fill. Sabe-se que a adesão dos sistemas adesivos aos substratos dentinários que foram submetidos previamente ao tratamento endodôntico é significativamente menor, por conta dos efeitos causados pelos irrigantes e substâncias químicas utilizadas durante o tratamento³⁹⁻⁴³.

Não restaurar adequadamente a porção coronária destruída, é um dos principais fatores que tem como consequência a extração dos dentes que foram endodonticamente tratados⁴⁴. Restaurar cavidades com compósitos Bulk-fill, especialmente após a conclusão do tratamento endodôntico, poderia significar praticidade e economia de tempo para os clínicos²⁶. Estudos vem sendo conduzidos para avaliar o grau de conversão^{45,46}, profundidade de polimerização^{32,47}, contração de polimerização^{33,48-51}, adaptação marginal^{19,31,35}, assim como propriedades mecânicas^{28,34,47,53}^{47,53,28,34}. No entanto poucos estudos avaliaram o desempenho das resinas compostas Bulk fill em dentes que foram tratados endodonticamente^{9,10,18,38,54}, e nenhum deles comparou o desempenho de resinas compostas Bulk-fill, que são indicadas para o selamento total das cavidades, utilizadas juntamente com os adesivos simplificados. A avaliação da viabilidade clínica desses novos materiais utilizados em conjunto faz-se necessária, uma vez que a sua utilização poderia representar uma otimização do procedimento restaurador com resinas compostas em dentes que foram tratados endodonticamente.

2 PROPOSIÇÃO

Esta pesquisa foi dividida em três artigos, tendo por objetivo:

1. Avaliar a adaptação interna de restaurações confeccionadas com resinas Bulk-fill e adesivos simplificados em dentes que foram tratados endodonticamente por meio da microscopia eletrônica de varredura;
2. Avaliar a resistência de união por meio do ensaio mecânico de microtração, entre a dentina tratada com agentes químicos utilizados durante o tratamento endodôntico e a associação entre adesivos simplificados e resinas Bulk-fill, de diferentes marcas comerciais, após 24 horas e após 1 ano;
3. Avaliar a resistência à fratura de dentes que foram tratados endodonticamente e restaurados com a associação entre adesivos simplificados e resinas Bulk-fill de alta viscosidade, de diferentes marcas comerciais.

A hipótese nula testada (H_0) é de que não há diferença entre os sistemas restauradores (Resinas Bulk-fill e sistemas adesivos all-in-one) avaliados quanto à adaptação interna, resistência de união (após 24 horas e após 1 ano) e resistência à fratura.

3 PUBLICAÇÕES

3.1 Publicação 1*

Research field focused on Dentistry

Origina Research

Title: Internal adaptation of bulk-fill composites restorations applied on RMGIC in endodontically treated teeth*

Giroto AC^a, Sanchez-Puetate CF^a, Piccioni MARV^a, de Andrade MF^a, Kuga MC^a

Abstract

The aim of this study was to evaluate the internal adaptation of several bulk-fill resins applied on a thin layer of RMGIC.

32 premolars were endodontically treated, filled with gutta-percha and cleaned. On the gutta-percha, a thin layer of resin-modified glass ionomer cement (RMGIC) Ionoseal (VOCO, GmbH, Cuxhaven, Germany) was applied. The teeth were divided into 4 groups (n=8): Control group (CG) Adper Scotchbond Multi-Purpose/Filtek Z350XT (3M/ ESPE); (FBF) Single Bond Universal/Filtek Bulk-fill (3M/ESPE); (SF) OptiBond All-in-one/SonicFill (Kerr), and (TBF) Tetric N-Bond Universal/Tetric N-Ceram bulk-fill (Ivoclar/Vivadent). Each tooth was sectioned, and replicas were analyzed with scanning electron microscopy (SEM). Dimensions of any existing gaps were measured using Image J software. Data were analyzed using ANOVA and Tukey's test ($p < 0.05$). The GAPS formed between the resins and the RMGIC and between RMGIC and dentin as well as the widths of GAPS were also evaluated.

TBF had the lowest percentage of GAPS and SF the highest ($p < 0.05$). In all groups, 100% of the specimens presented GAPS between RMGIC and dentin. The widths of GAPS between resin and dentin/RMGIC varied from 4.89 – 7.24 μm and between RMGIC/dentin varied from 22.81 – 26.88 μm .

There were significant differences between the resins studied in the present study, however, the GAPS between RMGIC and dentin should be considered more worrying.

Keywords: Adaptation; Bulk-fill composite; Dentin; Endodontics

* Artigo nas normas da Brazilian Oral Research (BOR).

Introduction

The effective coronal sealing is a fundamental for successful long-term of the endodontic therapy^{1,2}. If the root canal is exposed to saliva, the bacteria quickly migrate to the apex^{3,4}. Thus, the quality of the internal adaptation of the restorations can influence the bacterial recontamination of the root canal⁵. The materials that offer better coronal sealing are the composite resins used with adhesive systems^{6,7}, especially for cases with moderate loss of coronary structure⁸.

In endodontically treated teeth composite resin restorations are applied to deep cavities with high C-factor (ratio of bonded-to-unbonded restoration surfaces). One of the main problems of conventional composite resins is polymerization shrinkage⁹, which can have clinical consequences such as cracks in the dental structure, formation of gaps at the interface between the tooth and restoration, secondary caries, marginal staining and marginal microleakage^{10,11,12}. Conventional composite resins are applied with an incremental technique in an attempt to minimize these problems¹³. However, the application of multiple increments in these deep cavities consumes excessive clinical time^{14,15}.

Bulk-fill resins are indicated to solve the problems of conventional resins and optimize restorative procedures. According to manufacturers, these new composites can be polymerized at depths of up to 4–5 mm¹⁶, with low polymerization tension¹⁷. These resins are available in low-viscosity (also known as bulk-fill flow resins) or high-viscosity. Bulk-fill flow resins required an additional capping layer made of micro/nano hybrid layer of composite resin¹⁸. Few studies have evaluated the efficiency these materials in endodontically treated teeth^{16,18-24} and most of these studies evaluated only the fracture resistance^{16,18-21}.

During the polymerization reaction, the adhesive systems must resist the tensions generated, contributing significantly to the quality of the internal adaptation of the restorations²⁵⁻²⁷. As bulk-fill composite resins, all-in-one adhesive systems also are innovative materials, which offer simplified application, since they can be applied in a single operative step²⁸⁻³⁰, further optimizing the sealing of endodontically treated teeth esthetic restoration.

Since the high-viscosity bulk-fill resins are materials that optimize the restorative procedures in endodontically treated teeth, there are no studies that evaluate the internal adaptation in these teeth. Therefore, the aim of this study was to compare the internal adaptation of esthetic restorations, in endodontically treated teeth using

different high-viscosity bulk-fill resins, in conjunction with their respective all-in-one adhesive systems, on a thin layer of resin-modified glass ionomer cement (RMGIC). The null hypothesis tested is that there is no difference among the different restorative systems (bulkfill / adhesive resins) used.

Methodology

This study was approved by the Research Ethics Committee of Araraquara Dental School (#68339217.2.0000.5416) (ANEXO A). Thirty-two human maxillary premolars without caries, restorations, or root canal treatment that had been extracted for orthodontic reasons were used in this study. All teeth were cleaned and then stored in 0.5% Chloramine-T solution at 4 °C until use.

All the procedures were performed by 2 expert clinicians, respectively in endodontics and restorative dentistry. Tooth cusp were abraded using silicon carbide abrasive paper (grit No. 320) to obtain a polished occlusal surface. The coronary access was performed using a #FG3131 diamond bur (KG Sorensen; Cotia, SP, Brazil) at a high speed under water cooling. FG1801 PM.82 (KG Sorensen; Cotia, SP, Brazil) was placed parallel to the long axis of the tooth to obtain a standard cavity. Depth of the cavities was measured and the bur was replaced after preparation of five cavities. The final shape of cavity preparation was defined by the diamond bur shape. The dimensions of the preparation were confirmed using a periodontal probe.

Afterward, the working length was determined using a #10 K-file (Dentsply Maillefer; Ballaigues, Switzerland). Root canals were prepared using rotatory instruments file (ProTaper System; Dentsply Maillefer; Ballaigues, Switzerland) up to F3, at the working lengths. 2.5% NaOCl (Asfer; São Caetano do Sul, SP, Brazil) was used between instrument changes.

After chemical-mechanical preparation, each canal was irrigated with 3 mL of 17% EDTA (Biodinâmica; Ibioporã, PR, Brazil) for 3 min, and 10 mL of distilled water. In sequence, all root canals were dried using paper points (Dentsply-Herpo; Petropolis,RJ, Brazil). Root canals were obturated using F3 gutta-percha cones (Dentsply-Herpo; Petropolis, RJ, Brazil) and an epoxy-based sealer (AH Plus; Dentsply De-Trey, Konstanz, Germany), handled according to the manufacturer's instructions. The excess gutta-percha was removed, and the pulp chamber was cleaned with a cotton ball soaked in alcohol. A thin layer of resin-modified glass ionomer cement (RMGIC) Ionoseal® (VOCO GmbH; Cuxhaven, Germany) was applied on the gutta-

percha. All cavities were standardized with a 4 mm depth (Figura A1). The specimens were restored immediately after the end of endodontic treatment (Figura A2).

The specimens were divided into four groups (n=8) according to restorative materials (Table1) (Figura A1). The all-in-one adhesive systems were used in the self-etch technique. In all groups the adhesive systems and resins were from the same manufacturer. The application of all materials was done according to the manufacturer's recommendations.

(CG) Control Group: Scotchbond™ Multi-Purpose + Filtek™ Z350XT (3M/ESPE St. Paul, MN, USA). Acid etching was carried out using 35% phosphoric acid (Scotchbond Etchant Gel; 3M Espe, St. Paul, MN, USA), for 15 s on dentin. Three-step Scotchbond™ Multi-Purpose etch-rinse adhesive system was applied according to the manufacturer's instructions. Primer was applied for 20 s using a microbrush (KG Sorensen; São Paulo, SP, Brazil) and dried using a gentle air-spray for 5 s. Bond was applied and light-cured for 10 s using a LED-laser system (VALO™; Ultradent, South Jordan, UT, USA) with a 1000 mW/cm² output power. Filtek™ Z350XT nano-hybrid composite resin (shade A2, 3M ESPE; St. Paul, MN, USA) was inserted into the cavity using incremental technique. Restoration was performed using 2.5 increment with 2 mm thick oblique composite.

(FBF) Scotchbond™ Universal + Filtek™ Bulk Fill (3M/ESPE St. Paul, MN, USA). The Scotchbond™ Universal adhesive system was used as self-etch strategy. Adhesive was applied for 20 s, dried using a gentle air-spray and light-cured for 10s. Filtek™ Bulk Fill (shade A2, 3M ESPE; St. Paul, MN, USA) was inserted into the cavity with a 4mm thick single increment then light-cured for 20 s.

(SF) OptiBond™ All-in-one + SonicFill™ (Kerr Co; Orange, CA, USA). OptiBond™ All-in-one adhesive was used in self-etching strategy. Adhesive was applied using a microbrush for 20 s. A second adhesive application was carried out for 20 s then light-cured for 10 s. A 4 mm SonicFill composite single increment was placed using a SonicFill handpiece sonic-activation and light-cured for 20 s.

(TBF) Tetric®N- Bond Universal + Tetric®N-Ceram Bulk Fill (Ivoclar Vivadent AG, Schaan, Liechtenstein). Tetric®N- Bond Universal was used in self-etching strategy. Adhesive was rubbed for 20 s and light-cured for 10s. Tetric® N-Ceram Bulk Fill was inserted into the cavity with a 4mm thick single increment and light-cured for 20 s.

In all cavities, composite resin restorations were in contact with the RMGIC in the pulp wall and with the dentin in the proximal walls, similar to a clinical situation. The

specimens were stored in artificial saliva for 7 days at 37 °C. The restored teeth were cross-sectioned through their centers in a buccolingual direction, using a diamond saw (Isomet Diamond Wafering Blades, no. 11-4244, Buehler Ltd., Lake Buff, IL, USA) with water-cooling (Figura A3). The halves were polished using a sequence of silicon carbide abrasive papers (grits no. 1000, 1200, and 2000, Norton Abrasivos, Vinhedo, SP, Brazil) and felt disks containing 3, 2, 1, and 0.5 μ m diamond pastes (Buehler Ltd.). Specimens were sonicated (Thornton USC 1400, Unique Group, Indaiatuba, SP, Brazil) for 10 min to remove the polishing debris.

Impressions of the polished restored surfaces of the half were taken using a polyvinyl siloxane material (Express XT, 3M ESPE, St. Paul, MN, USA)(Figura A4) and were poured with an epoxy resin (EpoxiCure, Buehler Ltd.). The polymeric replicas were sputter-coated with gold to a thickness of approximately 50 Å, in a vacuum evaporator (SCD 050, Bal-Tec AG, Balzers, Liechtenstein) to analyze the presence of internal interfacial gaps in a scanning electron microscope (SEM) (JEOL, JSM-5600LV, Tokyo, Japan)²⁵(Figura A5).

For each specimen, approximately 24 images, each at a 200x magnification, were required to observe the entire length of the bonded internal interface. Image J software (National Institute of Health, Bethesda, MD, USA) was used to determine the length of individual debonded segments along the entire internal perimeter of the restoration. The SEM image scale bar was used for distance calibration. Individual lengths of debonded segments were obtained in millimeters, summed, and then converted to percentage of the total length of the interface. The GAPs formed between the resins and the RMGIC and between RMGIC and dentin as well as the widths of GAPs were also evaluated (Figura A6)(Figura A7).

Collected data were statistically analyzed using a 1-way analysis of variance test and Tukey post hoc test to evaluate differences in percentage of GAPs among groups. The significance level was set at 5% ($P < 0.05$).

Results

Table 2 shows the mean and standard deviation of the % GAPs between the composite resins studied and the dentin walls. The lowest % of GAP formation occurred in the TBF group ($P < 0.05$), with no statistically significant difference with the CG and Bulk-fill groups. The SF group had the highest percentage of GAPs ($P > 0.05$). The size of the formed gaps was not statistically difference between groups ($P > 0.05$) (Table 3) (Figure 1).

In 100% of the specimens evaluated, RMGIC was separated from the dentin walls (Figure 2). GAPS between RMGIC and dentin were much larger than those found between resins and dentin, regardless of the resin used (Table 3) (Figure 2 and 3).

All resins studied showed a perfect fit with RMGIC, except in the SF group where 50% of the samples formed GAPS between the RMGIC and resin (Figure 1).

Discussion

The null hypothesis that there is no difference in the internal adaptation between the different resins used was rejected, because bulk-fill SF resin showed the highest % of GAP formation, while the TBF showed the lowest. TBF has a high loading content and contains an initiator called Ivocerin, which has a higher photocuring activity than camphoroquinone. It can reduce the curing time and increase the curing depth of composites³². This bulkfill resin also contains low elastic modulus fillers that relieve shrinkage stress. Previous studies of adaptation have already found good results for this material^{31,33}.

No study had evaluated the internal adaptation of direct restorations in endodontically treated teeth. These cavities have a high C factor. The internal adaptation in cavities with a high C factor is inferior compared to with a low C factor¹³. Clinically after the gutta-percha is cut, a thin layer of resin modified GIC is applied^{18,34,35}, and on this layer the direct restorations are performed. In the 100% of the specimens evaluated, GAPS were found between this thin layer of RMGIC and resin, and they were much larger than those observed between the resins studied and dentin (Table 3, Figure 2).

Some studies have evaluated the use of RMGIC as liners in shallower cavities than the present study. As our results, they all found more GAPS when RMGIC was used³⁶⁻³⁸. On the other hand, some have suggested that dentin hybridization provides superior sealing³⁶. However, there are no study evaluating the effects of sealing directly on the gutta-percha.

The absence of GAP formation occurs due to the effective adhesion to the dentin, which resists the polymerization shrinkage^{39,40}. It has been suggested that the vectors of polymerization contraction indicate the occlusal surface when the resins are photopolymerized, resulting in debonding of the pulp floor composite^{40,41}. Therefore, the initial adhesion of the RMGIC was not strong enough to counterbalance the contraction force. The polymerization shrinkage of the composite resin tends to affect

the fragile interface between the RMGIC and the dentin, rather than affecting the interface between the RMGIC and the resin³⁸.

In our study, GAPs were found between the RMGIC and the dentin, but the RMGIC was perfectly adapted to the resin, except in the SF group where the formation of GAPs between these materials reached 50%. SF also showed the highest % of GAPs between resin and dentin. There is probably a relationship between these results, because in the other groups the good adaptation of the resin to the RMGIC may have caused a change in the stress distribution and thus contributed to the GAPs being located more in the ionomer than in the resins. However, more studies are needed to prove these results.

In previous studies, performed on Class II Cavity, SF showed good internal adaptation [13, 42]. The bulk placement of Sonic Fill System (Kavo, Kerr) is facilitated by a specialized handpiece, which delivers sonic energy at varying intensities. As sonic energy is applied through the handpiece, the incorporated modifier causes the viscosity to drop (up to 87%), during the composite insertion. When the sonic energy is stopped, the composite returns to a more viscous consistency. Because of these insertion characteristics, it was expected that these materials would present superior results of internal adaptation as in some previous studies^{33,42}. However, as in the study of Alquadaihi et al., 2018⁴³, the worst results of internal adaptation were found in this group. In addition, micro-ct images have found small bubbles within the material, and the authors speculated that these bubbles could be formed by resin vibration by Sonic energy³³.

High polymerization stress values are the main cause of GAPs formation along the tooth interface. However, a good-quality adhesive system is also important to maintain the union between the restoration and the walls of the tooth^{44,45}. In this study, prior to the application of the adhesive system, all teeth were submitted to endodontic treatment, which may affect adhesive efficiency⁴⁶⁻⁴⁹.

As the objective of the study was to evaluate simplified techniques for the sealing of endodontically treated teeth, adhesive systems were used in the self-etching technique. Adhesives from the same manufacturers as the evaluated resins were used. Despite the differences found among the resins, GAP formation did not exceed 30% on average in all experimental groups evaluated, a value that is small compared to other studies³¹. Thus, we can suggest that the endodontic treatment did not

drastically affect the effectiveness of dentin adhesion, which was relatively able to resisting the stress of polymerization.

Finding the best strategies to restore teeth that were treated endodontically is challenging, as many factors are involved such as: best material to apply on the gutta-percha, adhesive systems, and composite resins. The more simplified strategies were focused in this study, and despite providing interesting answers, this study has led to other questions to explore in future studies, such as evaluation of adaptation by different methods, evaluation strategies with ageing, questions regarding the use of RMGIC on gutta-percha, search for better materials for this purpose, and evaluate other adhesive systems and other bulk-fill resins.

Considering its limitations, this study concluded there was difference between the different high-viscosity bulk-fill resins used along with the universal adhesive systems. SF presented greater incidence of GAPs than TBF. However, the GAPs between RMGIC and dentin can be considered more worrying.

Compliance with Ethical Standards

Conflict of Interest

Author Giroto AC declares that she has no conflict of interest. Author Sanchez-Puetate CF declares that he has no conflict of interest. Author Piccioni MARV declares that she has no conflict of interest. Author de Andrade MF declares that he has no conflict of interest. And author Kuga MC declares that he has no conflict of interest.

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Ethical approval

This study was approved by the Research Ethics Committee of Araraquara Dental School (#68339217.2.0000.5416), all applicable international, national, and/or institutional guidelines for the care and use were followed.

Informed Consent

Informed Consent is not applicable.

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Tables

Table 1. Description of the composite resins used in the present study.

Composite Resin	Characteristics	Composition
Filtek^{MR} Z350 XT	Composite resin of incremental technique Maximum increment thickness: 2 mm Cor: A2 Maker 3M ESPE, St. Paul, MN, USA	Silane treated ceramics, silane treated silica, UDMA, Bis-EMA (6), Bis-GMA, silane treated zirconia, polyethylene glycol dimethacrylate, triethylene glycol dimethacrylate, 2,6-di-tert-butyl-p-cresol. Load 78.5% weight, 59.5% vol
FiltekTM Bulk Fill	Composite Resin Bulk Fill at low velocity Maximum increment depth: 5 mm Color: A2 Maker 3M ESPE, St. Paul, MN, USA	Bis-GMA, Bis-EMA, EBPADMA, TEGDMA, procrylate-based resin, camphorquinone, EDMAB, Zirconia, silica, ytterbium trifluoride, silanized ceramics. Load 77% weight, 59% vol
SonicFillTM	Composite Resin Bulk Fill at low velocity Maximum increment depth: 5 mm Color: A2 Maker: Kerr, Orange, CA, USA	Bis-GMA, TEGDMA, EBPADMA, SiO ₂ , glasses and oxides Load 86% weight, 66% vol
Tetric[®] N-Ceram Bulk Fill	Composite Resin Bulk Fill at low velocity Maximum increment depth: 4 mm Color: IVA Maker: Ivoclar Vivadent; Schaan, Liechtenstein	Bis-GMA, Bis-EMA, UDMA, barium glass, ytterbium trifluoride, mixture of oxides and prepolymers, additives, catalysts, stabilizers, pigments Load 78% weight, 55% vol (including prepolymer)

Bis-GMA, bisphenol A diglycidyl methacrylate; UDMA, Uretanodimethylmethacrylate; Bis-EMA (6), bisphenol A polyethylene glycol diether dimethacrylate; TEGDMA, triethyleneglycol dimethacrylate.

Table 2. Mean and standard deviation of the percentage (%) of GAPs (total GAP formation as a function of total interface length) of each experimental group.

	Mean (%)	Standard deviation
Control Group (CG)	13.58 ^{ab}	8.31
Filtek Bulk Fill (FBF)	17.19 ^{ab}	8.99
SonicFill (SF)	31.56 ^a	15.02
Tetric N-Ceram-Bulk Fill (TBF)	12.32 ^b	11.59

*Means followed by the same letters are not statistically significant ($p>0.05$).

Table 3. Mean (μm) and standard deviation (SD) of the widths of GAPs between RMGIC and dentin and between Resin and dentin.

	RMGIC/Dentin (SD)	Resin/Dentin (SD)
Control Group (CG)	22.81 ^a (12.61)	4.89 ^a (1.27)
Filtek Bulk Fill (FBF)	34.88 ^a (15.98)	6.89 ^a (3.94)
SonicFill (SF)	31.88 ^a (22.11)	7.24 ^a (2.80)
Tetric N-Ceram-BulkFill(TBF)	26.88 ^a (10.41)	5.94 ^a (3.8)

*Means followed by the same letters are not statistically significant ($p>0.05$).

Figures and Legends

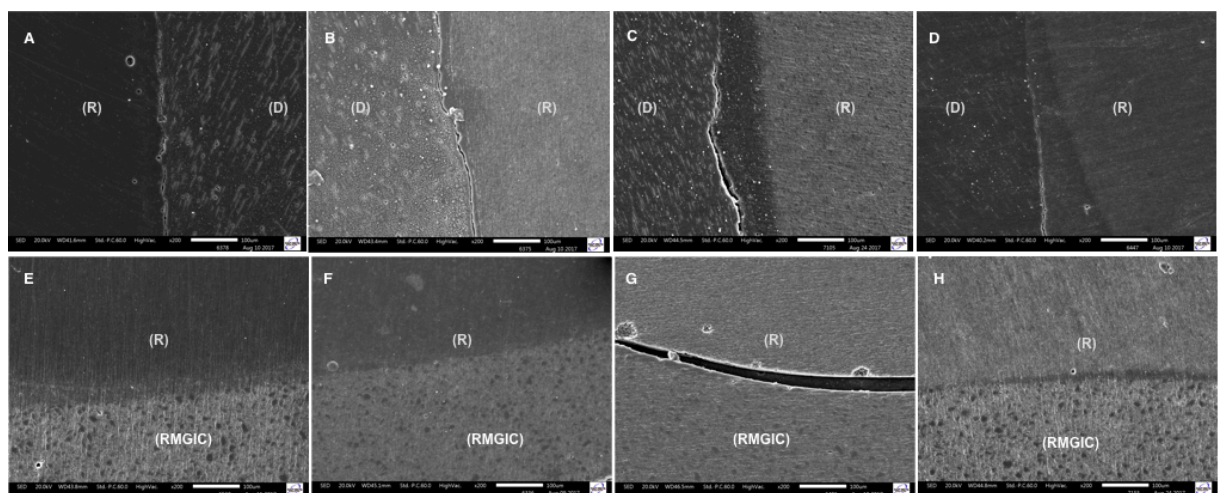


Figure 1. GAPs between the resins (R) and the proximal walls (D) (A, B, C, D). Adaptation between resins and RMGIC (E, F, G, H). A and E-Control Group (CG), B and F-Filtek Bulk-Fill (FBF), C and G-SonicFill (SF), and D and H -Tetric N-Ceram Bulk-Fill. (SF) was the only resin studied that presented GAPs with RMGIC.

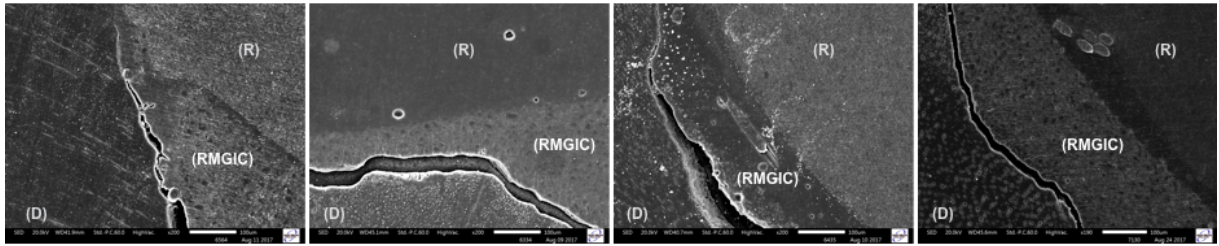


Figure 2. Examples of separation found between resin-modified glass ionomer cement (RMGIC) and dentin (D). In all cases, the perfect adaptation between the resin (R) and the (RMGIC) is noted.

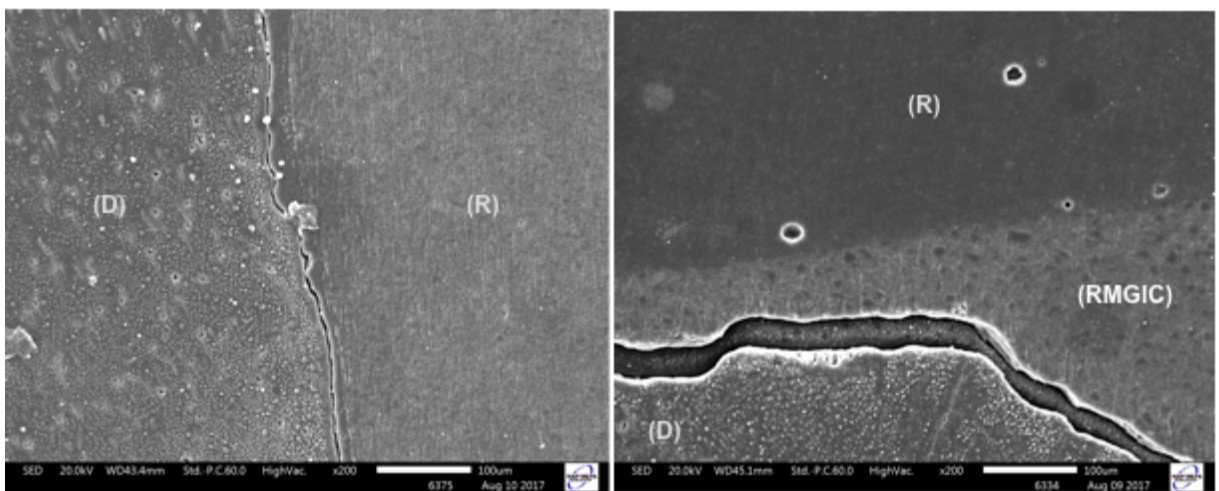


Figure 3. Difference between the gaps formed between the composite resin (R) and the dentin(D) and between resin-modified glass ionomer cement (RMGIC) and dentin (D).

3.2 Publicação 2*

Basic Research—Technology

Bond Durability of Simplified Adhesives Applied to Endodontically-Treated Dentin*

Abstract

Introduction: The aim of this study was to evaluate the microtensile bond strength (μ TBS) of simplified adhesive systems bonded to endodontically-treated dentin (ETD) after 24 h and 1 year of water storage. **Methods:** 40 human molars were cut to expose the deep dentin. The specimens were treated with 2.5% NaOCl for 30 min and 17% EDTA for 3 min and were then impregnated with AHPlus cement. After 5 min, they were then cleaned with 99% ethanol. The specimens were randomly divided into 4 groups according to the adhesive used: Scotchbond Multi-Purpose (SBMP; 3M/ESPE); Single Bond Universal (SBU; 3M/ESPE) – self-etch method; Optibond All-in-One (OPB; Kerr); or Tetric-N-Bond Universal (TBU; Ivoclar/Vivadent) – self-etch method. Resin blocks (Filtek Z350XT; 3M/ESPE) were constructed, specimens were stored in water at 37°C for 24 h, and the resin-dentin sticks from each tooth were prepared. After 24 h or 1 year, the μ TBS was measured. The adhesive interface was evaluated using confocal microscopy. Results were analyzed using two-way ANOVA with Tukey's test ($\alpha=0.05$). **Results:** No significant differences were observed among the adhesives after 24 h ($p>0.05$). However, significant differences were found after 1 year ($p<0.05$), just the SBU did not presented reduccion in μ TBS and were not significant different from the results obtained after 24 hours ($p<0.05$). The simplified adhesives OBA and SBU showed similar μ TBS values than the 3 step etch-and-rinse adhesive system SBMP ($p>0,05$).

Conclusions: The use of simplified adhesive systems on EDT produced immediate μ TBS was as efficient as 3 step-etch-and-rinse adhesive system. After 1 year, SBU maintained its original μ TBS. OBA and SBU showed similar μ TBS values than the 3 step etch-and-rinse adhesive system SBMP. SBU did not presented μ TBS reduction and TBU presented the worst results among the adhesive systems studied.

Keywords: Adhesives; Teeth, Endodontically-Treated; Dental Restoration, Permanent; Matrix Metalloproteinases

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Introduction

Today's adhesive systems available on the market can be classified into two categories: etch-and rinse (1,2) or self-etch strategies (1,3). Etch-and-rinse systems are available as two-step or three-step, while self-etching systems are available as one-step or two-step, one-step systems are also called all-in-one adhesive systems (2,3).

Previous studies demonstrated that the use of self-etch adhesives on dentin are beneficial for adhesive longevity (1,4). Because of the distinct characteristics of the substrate, etching on dental enamel with phosphoric acid results in better adhesive effectiveness (2,5). In order to allow the conditioning with phosphoric acid of enamel and the use of self-etch monomers on the dentin using a single bottle of adhesive, some manufacturers introduced the so-called universal adhesives systems. These adhesives provide versatility for the clinician to use both: etch and rinse or self-etch strategies (6,7).

After the endodontic treatment, the final restoration is critical for the prognosis of the tooth. (8-11). On the other hand, endodontically-treated dentin (ETD) is a challenging substrate for adhesion, for both etch-and-rinse adhesives (12,13) and self-etch adhesives (13-16). This challenge is the result of changes to the substrate caused by irrigants such as NaOCl (17-19) and EDTA (14,16,19), as well as by endodontic cements, since, even after cleaning with ethanol, these products leave remnants on the dental substrate (20,21).

The most simplified method for restore endodontically-treated teeth is through the use of universal adhesives and the self-etching method, or all-in-one self-etching adhesives. Few studies to date have compared the use of all-in-one adhesives on ETD to that of conventional adhesives (16). Of the studies that have evaluated the behavior of adhesive systems on ETD, none considered the effects of aging after storage (11-16,21). Meanwhile, in studies on normal dentin, most researchers have reported significant decreases in bond strength. This decrease is caused by the degradation of the adhesive tooth-composite interface, which occurs over time (1,22).

The objective of this study was to evaluate bond strength (μ TBS) and the hybrid layer formed by simplified adhesive systems (universal or all-in-one systems) on ETD both 24h and after 1 year by comparing it to the three-step etch-and-rinse adhesive system. The null hypothesis tested was that there would be no differences between the adhesive systems used on ETD.

Materials and Methods

1. Specimen Preparation

Forty cavity-free extracted human third molars were selected after the study was approved by the Research Ethics Committee on Research São Paulo State University (UNESP) Dental School in Araraquara São Paulo, Brazil. The teeth were cleaned and then stored in 1% chloramine T at 4°C. The enamel and surface dentin of the teeth were removed to expose the deep dentin (3mm above the cement-enamel junction) using a diamond disc at low speed (Isomet; Buehler Ltd., Lake Bluff, USA) under constant irrigation (Figura A8). The exposed dentin surfaces were then polished using 600-grit SiC paper for 60s under running water to create a standardized smear layer. The specimens were then treated in order to simulate the effects caused by substances used during the endodontic treatment. The teeth were individually immersed in 5 mL 2.5% NaOCl for 30 min, with the solution renewed every 3 min. They were then immersed in 5 mL 17% EDTA (Biodinâmica Ind. Com, Ibioporã, Brazil) for 3 min followed by a final washing with distilled water. The teeth were dried using absorbent paper. An epoxy resin-based sealer (AH Plus; Dentsply De Trey, Konstanz, Germany) was mixed in a 1:1 ratio of paste A and B, according to the manufacturer's instructions. The mixture was spread over the dentin surface using a microbrush (Microbrush Int, Grafton, WI, USA) until a visible sealer could be observed. The material was left undisturbed on the dentin surface of each specimen for 5 min. Finally, the dentin surface was cleaned using a cotton pellet saturated with 95% ethanol until the surface appeared visible clean (Figura A9).

2. Bonding Procedure

The specimens were randomly divided into four groups (n=10) according to the adhesive system used: Scotchbond Multi-Purpose (SBMP; 3M/ESPE); Single Bond Universal (SBU; 3M/ESPE) – self-etch method; Optibond All-in-One (OPB; Kerr); or Tetric-N-Bond Universal (TBU; Ivoclar/Vivadent) – self-etch method. The compositions, manufacturers, and application instructions of the four adhesives used in this present study are listed in Table 1. The adhesives were applied to the dentin surface and light cured for 10 s using a LED light curing unit set a 1000mW/cm² (VALO, Ultradent Products Inc., South Jordan, UT, USA).). After the bonding procedures, all teeth received a composite restoration. When simplified adhesives were used the restoration were built with a respective Bulk-fill composite of the same manufactures, then for Single Bond Universal Universal were used Filtek™ Bulk-fill (3M ESPE, St.

Paul, MN, USA), for OptiBond® All-in-one were used SonicFill™ (Kerr, Orange, CA, USA) and for Tetric® N-Bond Universal were used Tetric® N-Ceram Bulk-fill. This restorations were made with a single increment of 4mm each, and light cured for 20s (VALO®, Ultradent Products Inc., South Jordan, UT, USA). For Scotchbond Multi Purpose were used nanofilled composite resin (Filtek Z350XT, 3M ESPE, St. Paul, MN, USA) in to increments of 2mm each. Each increment was light cured for 20s under the LED light curing mentioned above.

After storing in distilled water for 24 h at 37°C, the specimens were then longitudinally sectioned in mesial-distal and buccal-lingual directions across the bonded interface using a slow-speed diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) to obtain resin-dentin sticks with a cross-sectional area of approximately 1 mm² as measured with a digital caliper (Digimatic Caliper, Mitutoyo, Tokyo, Japan). One hundred and thirty-seven sticks were obtained from each group. Half of the sticks were used immediately (after 24 h) for the μ TBS, while the remaining half were used after 1 year of storage in distilled water.

3. Microtensile Bond Strength (μ TBS) Testing

For μ TBS testing, the resin-dentin sticks were fixed to a testing jig with cyanoacrylate glue (Super Bonder gel; Loctite Henkel, Rocky Hill, CT, USA) and tested to failure using a universal test machine (EZ-test; Shimadzu, Kyoto, Japan) at a crosshead speed of 1 mm/min. The μ TBS values (MPa) were calculated by dividing the load at failure by the cross-sectional bonding area.

The failures mode of the specimens was classified as cohesive (failures exclusive within dentin or resin composite), adhesive (A; failure at the resin-dentin interface) or mixed (failures at the resin-dentin interface, which included cohesive failure of neighboring substrates. The classification was performed unde a stereo microscope at 60x magnification (Olympus SZ40, Tokyo, Japan).

4. Confocal Laser Scanning Microscopy Analysis (CLSM)

Rhodamine B fluorescent dye (Merck, Darmstadt, Germany) at a concentration of 0.01 wt% was added to the bonding agent prior to application to the dentin surface. Using the same protocols described above, three specimens per group were prepared for observation under a confocal laser scanning microscope. Each sample divided in half (for analyses after 24 h and 1 year of storage), and both surfaces were wet-polished for 1 min with SiC paper (Grit 800/1200/2000). The samples were immersed

for 3 h in a solution of Sodium Fluorescein 100 mM before assessment using a Leica SP5 TCS Confocal laser scanning microscope (Leica Microsystems, Hanheim, Germany). Finally, 20 μ m of Z-stack was assessed at a 0.2 μ m step size.

5. Statistical Analysis

Data from μ TBS were analyzed using two-way ANOVA and Tukey's post-hoc test at ≤ 0.05 .

Results

The μ TBS results are summarized in Table 2. Immediately (24 h) after the procedure, there were no statistically significant differences in μ TBS produced by the adhesive systems studied ($p>0.05$). However, significant differences were found after 1 year ($p<0.05$), just the SBU did not presented reduccion in μ TBS and were not significant different from the results obtained after 24 hours ($p<0.05$). The simplified adhesives OBA and SBU showed similar μ TBS values than the 3 step etch-and-rinse adhesive system SBMP ($p>0,05$). TBU adhesive systems exhibited lowest μ TBS after 1 year and was significant diferent from the SBU and OPB ($p<0.05$).

Figure 2 shows representative CLSM images of the adhesive interfaces from each experimental group. The hybrid layer formed by SBMP is clearly identified by the high concentration of resin tags in the dentinal tubules. After 1 year, signs of degradation are visible and are accompanied by the presence of water in the hybrid layer. Out of all of the adhesive systems, the adhesive layer of the SBU remained the most stable after 1 year of aging. The specimens on which the OPB adhesive was used were also found to have water in the hybrid layer after 1 year. Fractured tags were found in the TBU specimens tested after 24 h and after 1 year.

Discussion

The null hypothesis tested was rejected, there was no difference between the results produced by the adhesive systems after 24 h, the specimens did differ based on the materials evaluated after 1 year. Our results show that, in addition to the lack of statistically significant differences in immediate μ TBS values, all of the adhesives were found to penetrate the dentin well. It is known that micromechanical retention to dentin is essential for immediate μ TBS. Good penetration was expected in the case of SBMP, since in this system was used phosphoric acid, which completely removes the smear layer and demineralizes the dental surface, exposing the collagen fibrils so that they can be infiltrated by the hydrophilic monomers present in the primer (1,2).

The interaction between normal dentin and self-etching adhesives is more superficial (3,23), a result which differed from that which was observed in our confocal microscopy images. The aggressiveness of the self-etching adhesives, which depends on pH levels, may change the depth of interaction of these adhesives (24). According to the manufacturer, the pH of the adhesives used in this study ranges from 2.5 to 3 (Table 1); the adhesives are therefore classified as ultra-mild. Ultra-mild self-etching adhesives tend to form one thin sub-micrometric hybrid layer, and resin tags are infrequent (23).

Adhesives with a weaker pH may exhibit better penetration into endodontically-treated dentin due to the increase in permeability caused by NaOCl (25) and EDTA (26), which likely create a smear layer that differs from those which are found in substrates without endodontic treatment. The good level of penetration exhibited by all of the adhesives studied may be associated with the good immediate bond strength results produced by these materials. Furthermore, the hybrid layer pattern may be explained by the fact that the substances used during the endodontic treatment were applied to the deep dentin. In deep regions, the density of the tubules is known to be approximately 45,000 per μm^2 and they are approximately 2.37 μm in diameter; the area occupied by intertubular dentin is 12% (27).

However, even the endodontic sealer (20,21) and EDTA (14,16,19) contributing to reduce immediate μTBS , NaOCl is considered the main agent responsible for these differences, since it interferes with the polymerization of the adhesive materials (28). Free radicals remain on the dentin and compete with the propagation of vinyl free radicals generated during the photoactivation of the adhesive systems, which results in a premature termination of the chain and incomplete polymerization (12,29).

It is for these reasons that most studies consider the immediate effects of adhesive systems (12-15). However, long-term efficacy may be influenced by adhesive composition. According to our results, the simplified adhesives tested herein exhibited inconsistent, material-dependent behaviors, since some adhesives diminished bonding performance over time while others did not.

During the application of the 3 step etch-and-rinse system (SBMP), adequate moisture levels must be maintained to prevent the collapse of the collagen fibrils, which may decrease μTBS (2). Due to the degree of concentration, when the resin monomers infiltrate the demineralized collagen matrix, some regions are not adequately filled in, which produces defects inside the hybrid layer (30). Under these conditions, water-

filled niches activate endogenetic enzymes present in the collagen matrix (31), and these enzymes trigger hydrolytic and enzymatic degradation that negatively affects the long-term efficacy of the adhesive (32).

As is the case with normal dentin (31,33,34), the exposed collagen of endodontically-treated dentin was found to be vulnerable to hydrolytic degradation processes after 1 year of storage. The confocal microscopy images prove this phenomenon by showing water within the adhesive layer on the SBMP and OPB.

SBU was the only adhesive that did not presented reduccion in μ TBS, the results after one year were not significant different from the results obtained after 24 hours. Furthermore, it also differed from the SBMP and OPB adhesives in that water was not found in the hybrid layer after aging. The composition of this adhesive includes the functional monomer 10-Methacryloyloxydecyl dihydrogen phosphate (10-MDP). Relative to other monomers, 10-MDP possesses a longer hydrophobic spacer chain, which results in intense and stable adhesion, and it has been found to contribute to bond durability and to protect the hybrid layer from hydrolytic degradation (37,38). The results of other studies on normal dentin are consistent with our findings (39).

The functional monomer in OPB is glycerol-phosphate dimethacrylate (GPDM). A recent study concluded that the bond formed between GPDM and hydroxyapatite was weaker than the strong bond formed between hydroxyapatite and MDP. Thus, though this adhesive contains acetone, which is a solvent that contributes to long-term efficacy, the lack of formation of a stable calcium salt resulted in diminished μ TBS after aging (38). The confocal microscopy images revealed the presence of water within the hybrid layer. However, even that the μ TBS values have diminished after 1 year, there were not statistical differences between this adhesive and the adhesives SBMP and the SBU.

The mixture and dilution of 10-MDP with other monomers to produce new adhesive products, as well as the concentration of this monomer in different adhesive systems, may impact the ability of 10-MDP to provide adhesive stability (39). The Tetric N-Bond Universal adhesive contains both 10-MDP and the functional methacrylated carboxylic acid polymer (MCAP), which is also capable of reacting with and bonding to hydroxyapatite. This fact may explain the weaker μ TBS after 1 year, since 10-MDP and MCAP may have competed in their bonding with the calcium of the dentin. Furthermore, differences in purity of MDP from different manufacturers may result in

different adhesive behaviors (40). The confocal microscopy images of this study obtained immediately (after 24 h) and after 1 year of storage revealed fractured tags. Our studies show that more research is necessary to consider the behaviors of other adhesive systems applied to ETD. Future research must consider aging and the hybrid layers formed.

Within the limitations of this study, we can conclude that the use of simplified adhesive systems on ETD produced immediate adhesive outcomes that were as effective as those produced by a three-step etch-and-rinse adhesive system. After 1 year, the composition in the SBU adhesive system was able to maintain the same bond strengths found 24 h after the procedure, and this adhesive system produced superior results relative to the other adhesive systems tested. This adhesive system was the only one that did not presented statistically difference between the μ TBS values obtained immediately (after 24 h) and after 1 year of storage. SBU and OBA showed similar μ TBS values than the 3 step etch-and-rinse adhesive system SBMP. TBU presented the worst results between the adhesive systems studied and was statistically different from the SBU and OPB.

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The authors deny any conflicts of interest.

Statement of Clinical Relevance (40 words)

Use of simplified adhesive systems on endodontically treated dentin can optimize restorations after root canal treatment. After 1 year, composition in the Single Bond Universal could be responsible to maintain the same bond strengths found 24h after the procedure.

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Figure 1. Lists the distribution of the failures modes based on stereomicroscopic observation. In all groups, the failures modes were primarily adhesive/ mix failure.

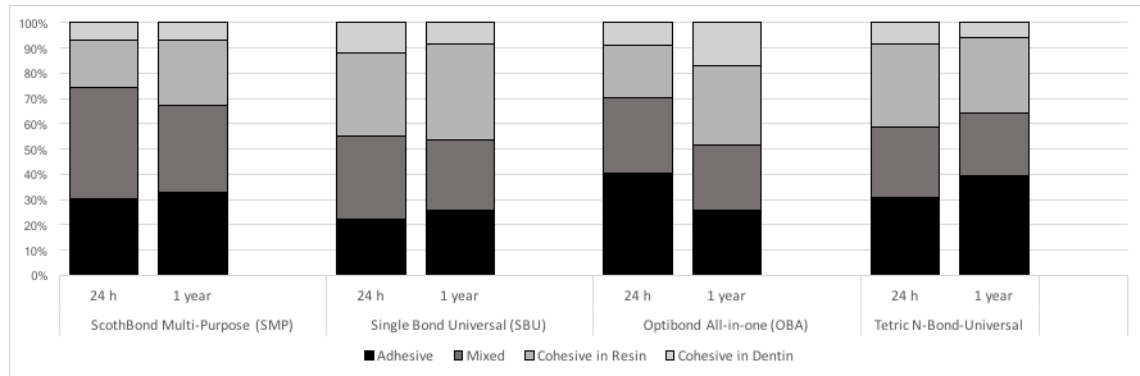
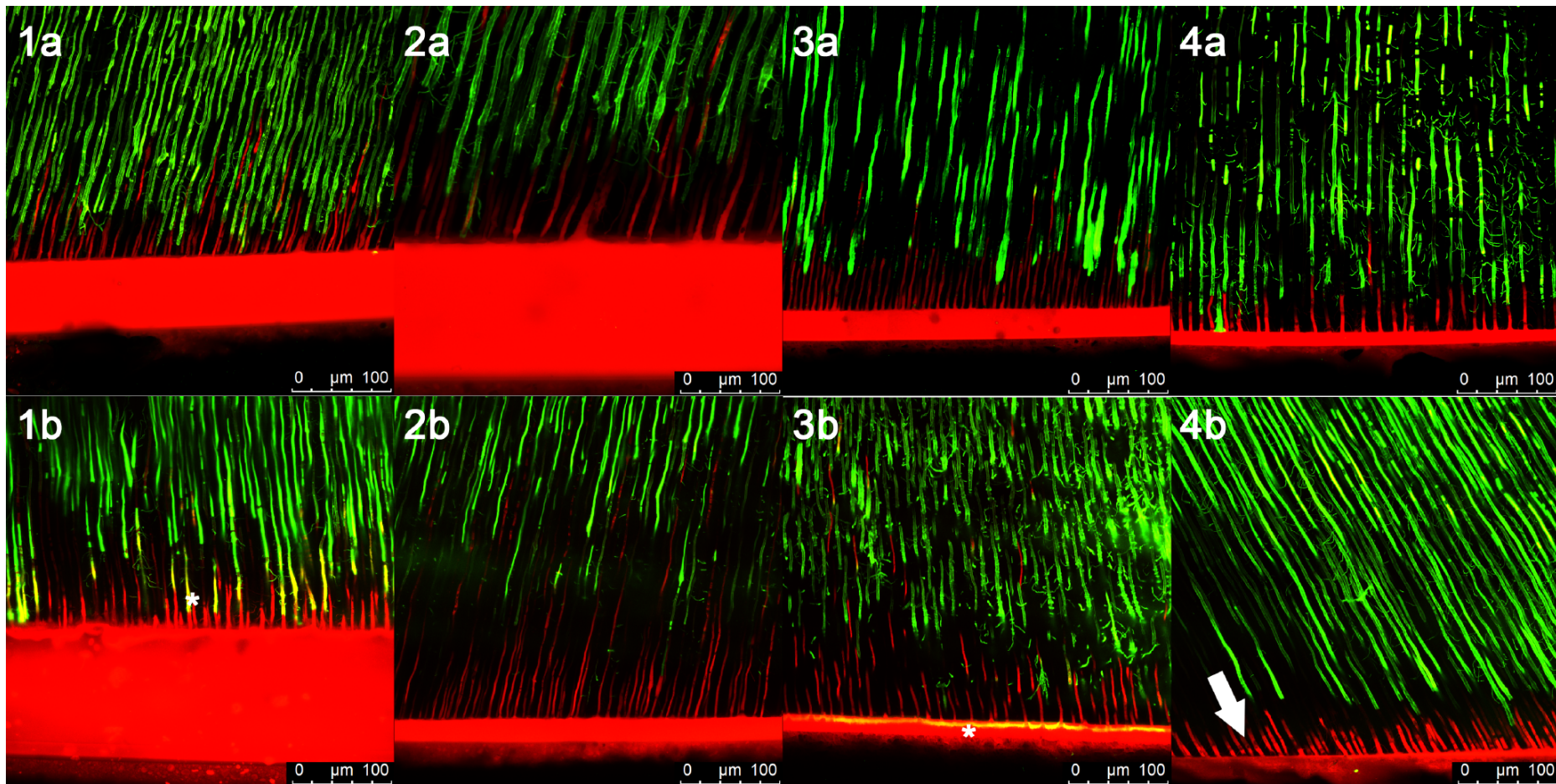


Figure 2. Representative confocal laser scanning micrographs (CLSM) images of the resin-dentine adhesive interfaces of each experimental group. Adhesive 1 (SBMP), 2 (SBU), 3(OPB) and 4 (TBU). Letter a (24hr) and b (After 1 year). The arrow points the resin tags fractured within the hybrid layer. The asterisk the infiltration in yellow of water within the adhesive layer.



Tables

Table 1. Adhesive system (Manufacturer), Composition and Application mode.

Adhesive system (Manufacturer)	Composition ^a	Application mode ^b
Scotchbond Multi-Purpose (3M/ESPE, St. Paul, MN, USA) 3 step etch-and-rinse adhesive system	<u>Universal Etchant</u> : water, 32% phosphoric acid, synthetic amorphous silica, polyethylene glycol, aluminium oxide <u>PRIMER</u> : HEMA, polyalkenoic acid methacrylate copolymer, water <u>BOND</u> : Bis-GMA, HEMA, dimethacrylates photoinitiators.	Etch-and-rinse strategy 1. Acid etching (15s), rinsing (15s) and drying leaving dentin moist 2. Application of 2 coats of primer; 3. Air-dry (10s at 20cm) 4. Application of the adhesive; 5. Light-activation for 10s
Single Universal Adhesive (3M/ESPE, St. Paul, MN, USA) Universal adhesive pH 2,7	MDP phosphate monomer, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane	Self-etch strategy 1. Apply the adhesive to the entire preparation with a microbrush and rub it for 20s; 2. Direct a gentle stream of air over the liquid for about 5s until it no longer moves and the solvent has evaporated completely; 3. Light polymerize for 10s.
OptiBond® All-In-One (Kerr Corporation, Orange, CA, USA) 1 step self-etch adhesive system (All-in-one) pH 2,5	GPDM, HEMA, GDMA, Bis-GMA, water, acetone, ethyl alcohol, camphorquinone, nanosilica and sodium hexafluorosilicate fillers	1. Adhesive application and scrubbing for 20s (two layers); 2. Gentle air blow, then medium-force air dry for 5s; 3. Light cure for 10s.
Tetric® N- Bond Universal (Ivoclar-Vivadent, Schaan, Liechtenstein) Universal adhesive pH 2,5 – 3.	HEMA, MDP, Bis-GMA, MCAP, D3MA, ethanol, water, highly dispersed silicon dioxide and camphorquinone	Self-etch strategy 1. Scrub one coat of adhesive for 20s; 2. Gently air thin for 5s; 3. Light-cure for 10s at 1000mW/cm ²

a. HEMA, 2-hydroxyethyl methacrylate; Bis-GMA, bisphenol glycidyl methacrylate MDP, methacryloyloxydecyl dihydrogen phosphate; GPDM, glycerol phosphate dimethacrylate; MCAP, methacrylated carboxylic acid polymer; D3MA, decandiol dimethacrylate

b. According to the manufacturer's instructions

Table 2. Means (standard deviations), of μ TBS results after 24h and 1 year.

Sistemas Adesivos	24 h	After 1 year
(SBMP) Scotch Bond Multi - Purpose	51,40 (9,82) ^{Aa}	39,66 (6,94) ^{ABb}
(SBU) Single Bond Universal	54,03 (10,21) ^{Aa}	45,35 (8,06) ^{Aa}
(OPB) OptBond All-in-one	54,42 (13,59) ^{Aa}	41,73 (3,18) ^{Ab}
(TBU) Tetric-N- Bond Universal	54,55 (9,83) ^{Aa}	31,30 (8,67) ^{Bb}

Means followed by the same letter (capital letters in the columns and lower letters in the rows) indicate statistically similar values($p>0.05$).

Capital letters in the columns compare different adhesives ($p>0.05$). Lower letters in the rows compare different times ($p>0.05$).

3.3 Publicação 3*

Title: Fracture resistance of endodontically treated teeth restored using different high-viscosity bulk fill composites*

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Abstract

Introduction. Endodontically treated teeth are more susceptible to fracture than vital teeth. Composite restoration can improve the endodontically treated teeth fracture resistance. Moreover, it improves tooth sealing, function, aesthetics, marginal sealing, and remnant dental protection.

Objective. The aim of this *in vitro* study was to evaluate the fracture resistance of endodontically treated teeth restored using different high-viscosity bulk-fill composites.

Material and Methods. Fifty sound human maxillary premolars were subject to endodontic treatment. Teeth were randomly divided into 5 groups (n=10) according to bulk fill composite: positive control Scotchbond™ Multi-Purpose + Filtek™ Z350XT (PCG); negative control, no restoration, (NCG); Scotchbond™ Universal + Filtek™ Bulk Fill (FBF); OptiBond™ All-in-one + SonicFill™ (SF) and Tetric®N- Bond Universal + Tetric®N-Ceram Bulk Fill (TBF). All composites were used with their respective adhesive system. Universal adhesive systems were used as self-etch strategy. The restored teeth were stored in distilled water for 24 h. Specimens were subjected to a fracture resistance test at a 0.5 mm/min speed in an electromechanical testing machine. Fracture resistance and failure mode were recorded. Data were subjected to one-way ANOVA and Tukey tests ($\alpha=5\%$).

Results. Endodontically treated teeth restored using composite resin presented similar fracture resistance ($p<0.05$). The lowest fracture resistance value was observed in non-restored teeth (NCG) ($p<0.05$).

* Artigo submetido nas normas do Iranian Endodontic Journal.

Conclusion. Endodontically treated teeth restored using high-viscosity bulk-fill composites presented similar fracture resistance to those teeth restored with conventional nanohybrid composite.

Keywords Bulk-fill composites. Endodontically treated teeth. Fracture resistance. Root filled teeth, composite resin, Fracture strength, Fracture pattern.

Introduction

The success of endodontic treatment depends on good endodontic therapy and an adequate coronary restoration after root canal obturation. Dental crown can be weakened by caries, crown fracture, endodontic procedures, and cavity preparation. Endodontically treated teeth are more susceptible to fracture than vital teeth ^{12,15} due to the moisture and anatomic structures losses. A clinical study has shown that presence of non-restorable cavities and fractured cusps were responsible for the endodontically treated teeth extraction presenting a 17.8% incidence ^{17,18} reported in factors related to the endodontically treated teeth extraction after periodontal disease and endodontic treatment failures, and concluded that damages caused by cavities and fractures are the main factors for the endodontically treated extraction.

Composite restorations can improve the endodontically treated teeth fracture resistance¹⁴. Moreover, coronary restorations improve tooth sealing, function, aesthetics, marginal sealing, and remnant dental protection¹⁰. Many restorative materials using bulk fill are available in dental industry, however, no studies have evaluated their long-term success in endodontically treated teeth ¹. Conventional composite resins are normally used in endodontically treated teeth restoration, however the polymerization shrinkage is still a problem. Polymerization shrinkage creates high stress along the tooth/restoration interface resulting in clinical complications, and increasing dental fracture risks². Incremental technique has been used to reduce shrinkage stress and promote a proper polymerization depth. Nevertheless, 2mm thick increments technique requires longer clinical time, increasing the risk of contamination between increments, and may cause empty bubbles incorporation^{1,3}.

Bulk fill composites have been introduced as a concept restorative material in dental industry in order to minimize the incremental technique errors and reduce restoration clinical time¹⁶. According to the bulk fill manufacturer', those materials can

be efficiently light-cured in depths up to 4-5mm with low stress of polymerization. Bulk fill composites are classified as *low-and-high-viscosity* formulations. Low-viscosity bulk fill composites, also called flowable bulk-fill, require a final conventional composite resin layer to cover bulk fill materials as they present low surface hardness and modulus of elasticity. On the other hand, high-viscosity bulk fill resin composites, also called sculptable or condensable, are more resistant to slumping and contain greater filler load, thus, sculptable materials may be inserted in a single-step application into the cavity without any composite covering^{6,11}.

One of the main indications of endodontically treated teeth extraction is the absence of a proper coronary restoration⁴. In addition, bulk-fill composites can optimize clinical time and be more practical for clinicians after endodontic treatment. However, few studies have been conducted to analyze endodontically treated teeth restored using bulk-fill composites^{1,5,8,13,19,25}. Not a great number of studies have evaluated the fracture resistance of endodontically treated teeth restored using bulk-fill flowable composites^{1,5,8,19,25}. However, no study has evaluated the fracture resistance of endodontically treated teeth restored using different high-viscosity bulk-fill composites.

Therefore, the aim of this study was to evaluate the fracture resistance of endodontically treated premolars restored with different high-viscosity bulk-fill composites with their universal adhesive system as a self-etching strategy. The null hypothesis was that high-viscosity bulk-fill composites do not improve the fracture resistance of endodontically treated premolars.

Material and Methods

Specimen pre treatment

This study was approved by the Research Ethics Committee of Araraquara Dental School (#68339217.2.0000.5416). Fifty sound human maxillary premolars with completely formed apices extracted by orthodontic reasons were selected. The inclusion criteria required teeth with similar crown size and root dimensions. The teeth were evaluated using a digital calliper (Series 480-505, resolution 1 μ , SHAN, Precision Measuring Instruments, Guilin, China), stereomicroscope (Leica Microsystems, Biberach, Germany) at a 20x magnification, and radiographic analysis. Exclusion criteria excluded specimens with cracks or anatomical anomalies, root canal treatment and restoration. Soft tissues and calculus were mechanically removed using

periodontal curette. After that, specimens were kept in a 0.5% Chloramine-T solution at 4°C until use.

Buccal-lingual and mesio-distal average widths were 14.5 +/- 1mm x 8 +/-1 mm, respectively. A wax layer (0.2-0.3 mm) was applied to the external root surface of each tooth. The tooth roots were inserted in a PVC cylinder (3cm length x 2cm internal width) embedded in polyester resin (Maxi Rubber, São Paulo, Brazil), placed 1mm below the cement-enamel junction (CEJ). The whole set rested for 24 h until the resin polymerization was completed. Then the root surface wax was melted using boiling water, and the space was filled with polyvinylsiloxane impression material (Oranwash vl, Zhermark, Germany) simulating the periodontal ligament.

Cavity preparation

A single operator performed the cavity preparations and another one carried out the endodontic treatment. The coronary access was performed using a #FG3131 diamond bur (KG Sorensen, Cotia, Brazil) at a high speed under water cooling. FG1801 PM.82 (KG Sorensen, Cotia, Brasil) was placed parallel to the tooth long axis in order to obtain a standard cavity. Cavities depth were measured and the bur was replaced after five cavities preparation. The final shape of cavity preparation was defined by the diamond bur shape. The preparation dimensions were measured using a periodontal probe.

Root canal treatment

Afterward, the tooth working length was determined using a #10 K-file (Dentsply Maillefer, Ballaigues, Switzerland). Root canals were prepared using ProTaper rotatory instruments file (Dentsply Maillefer, Ballaigues, Switzerland) up to size #30 (F3), at the working lengths. 3mL of 2.5% NaOCl (Asfer, São Caetano do Sul, SP, Brazil) was used between instrument changes. After biomechanical preparation, each canal was irrigated with 3mL of 17% EDTA (Biodinâmica, Ibiporã, Brazil) for 3 min, irrigated with 10mL of distilled water and then dried using paper points (Dentsply-Herpo, Petropolis, Brazil). Root canals were obturated using F3 gutta-percha cones (Dentsply-Herpo, Petropolis, Brazil) and an epoxy-based sealer (AH Plus, Dentsply De-Trey, Konstanz, Germany), handled according to the manufacturer's instructions. After obturation, a heated plugger was used to remove the coronal gutta-percha, with no further vertical compactation. Radiographs were taken to evaluate the obturation quality. A heated plugger was used to remove 3mm of gutta percha from the root canal, and a cervical barrier with a light-curing glass ionomer (Ionoseal, VOCO GmbH, Cuxhaven,

Germany) was placed in the cervical radicular. All cavities were standardized with a 4mm depth.

Restorative procedure

The specimens were divided into five groups (n=10) according to restorative materials (Table 1), as following:

(PCG) Positive Control Group: Scotchbond™ Multi-Purpose + Filtek™ Z350XT (3M/ESPE St. Paul, MN, USA). Acid etching was carried out using 35% phosphoric acid (Scotchbond Etchant Gel, 3M Espe, St. Paul, MN, USA) for 15 s in dentin . Three-step Scotchbond™ Multi-Purpose etch-rinse adhesive system was applied according to the manufacturer's instructions. Primer was applied for 20s using a microbrush (KG Sorensen, São Paulo, Brazil) and dried using a gentle air-spray for 5 s. Bond was applied and light-cured for 10s using a LED-laser system (VALO™; Ultradent, South Jordan, UT, USA) with an 1000mW/cm² output power. Filtek™ Z350XT nano-hybrid composite resin (shade A2, 3M ESPE, St. Paul, MN, USA) was inserted into the cavity using incremental technique. Restoration was performed using 2,5 increment with 2mm thick oblique composite.

(NCG) Negative Control Group: No restoration.

(FBF) Scotchbond™ Universal + Filtek™ Bulk Fill (3M/ESPE St. Paul, MN, USA). The Scotchbond™ Universal adhesive system was used as self-etch strategy. Adhesive was applied for 20s, dried using a gentle air-spray and light-cured for 10s. Filtek™ Bulk Fill (shade A2) was inserted into the cavity with a 4mm thick single increment then light-cured for 20s.

(SF) OptiBond™ All-in-one + SonicFill™ (Kerr Co, Orange CA, USA). OptiBond™ All-in-one adhesive was used as self-etching strategy. Adhesive was applied using a microbrush for 20 s. A second adhesive application was carried out for 20 s then light-cured for 10 s. A 4mm SonicFill composite single increment was placed using a SonicFill handpiece sonic-activation and light-cured for 20 s.

(TBF) Tetric®N- Bond Universal + Tetric®N-Ceram Bulk Fill (Ivoclar Vivadent AG, Schaan, Liechtenstein). Tetric®N- Bond Universal was used as a self-etching strategy. Adhesive was rubbed for 20s and light-cured for 10s. Tetric® N-Ceram Bulk Fill was inserted into the cavity with a 4mm thick single increment and light-cured for 20 s.

The specimens were stored in artificial saliva for 24 h at 37°C.

Fracture resistance test

The specimens were placed in a fixed device and subjected to a fracture resistance test using a 5kN load cell at a 0.5mm/min speed in an electromechanical testing machine (EMIC DL 2000; São José dos Pinhais, Brazil). The last force applied before the crown fracture was determined.

Failure mode

The fractured specimens were examined using stereomicroscope (40x) to evaluate the failure mode have classified as: Repairable fractures occurred above the simulated bone level (1mm from apical to cement-enamel junction) and were defined as “Favorable failures”, whereas, unrepairable fractures below the simulated bone level were defined as “Unfavorable failures”.

Statistical analysis

Data were subjected to a one- way ANOVA followed by post hoc Tukey test at a 5% significance level. Failure mode data were submitted to Chi-square test ($p < 0.05$).

Results

Fracture resistance test

Fracture resistance mean values (N) and standard deviations are described in Table 2. Negative control group (NCG, 215.14N) presented the lowest values ($p < 0.05$). No statistically significant differences were found among the restored groups (PCG, FBF, SF and TBF) ($p > 0.05$). All groups presented values higher than negative control group.

Failure mode

Table 3 illustrates failure mode frequency among groups. 54% failures mode were favorable. Positive control and FBF groups presented more favorable fractures, while negative control and SF groups presented more unfavorable fractures.

Discussion

The present study has noticed that no fracture resistance difference was found among endodontically treated premolars restored using a high viscosity bulk-fill composites (Filtek Bulk fill, SonicFill and Tetric-N-Ceram Bulk-fill) or a conventional nano-hybrid resin composite (Filtek Z-350 XT). Therefore, the null hypothesis should be accepted.

Unrestored teeth became progressively weaker with more extensive preparations⁵. Dentin preservation is relevant to dental integrity maintenance and restored endodontically teeth prognosis¹⁸. According to Reeh, et al.¹² any endodontic

procedure presents a small effect on the tooth, reducing its relative rigidity by 5%, mainly caused by coronary access opening. Thus, this study aimed at evaluating the restorative materials sealing with the smallest coronary access. Other studies have evaluated bulk fill composites performance mainly in Class II cavities ^{1,8,19}.

However, no studies have analyzed it using high-viscosity bulk fill resins similarly to the present study. Atalay, et al.¹ (2016), compared the fracture resistance of endodontically treated teeth, Class II MOD cavities, restored with different composites (high-viscosity bulk fill, flowable bulk fill, fiber-reinforced, and conventional composite resin) similarly to this study, and it did not find any significant differences among the restorative materials.

Many studies have been conducted to determine the best restorative materials once the restoration quality is relevant to the endodontically treated teeth prognosis ^{1,8,19}. High-viscosity bulk-fill resins associated with universal adhesives are more practical, reducing the clinical time, providing comfort for patients. The present study suggests that coronary access sealing using those materials is efficient since fracture resistance was similar among restored groups. In addition, some studies have shown that high-viscosity bulk fill composites present further advantages, such as high filler content (wt/vol%), low polymerization shrinkage and shrinkage stress in comparison with flowable bulk-fill composites ⁷. Han & Park ⁷ have reported that flowable bulk fill composite can demonstrate inferior internal adaptation in comparison with high viscosity bulk-fill composite.

Failure mode classification considered the depth, extent and restorative fractured teeth difficulty. Thus, failures were divided into favorable and unfavorable. Isufi, et al.⁸ (2016) showed that more than 70% of restored teeth presented unfavorable fractures, and the study attributed the fractures severity due to cervical dentin removal.

Universal adhesives associated to high viscosity bulk-fill composites can simplify the endodontically treated teeth sealing. No study has associated universal adhesives as self-etching strategy to bulk fill composites in endodontically treated teeth. Regarding the fracture resistance, the high viscosity bulk-fill composites and universal association were favorable as they did not reduce the restored teeth mechanical resistance, while facilitating the restorative procedure with reduced clinical time.

Within the limitations of this *in vitro* study, it used a static load cell causing the teeth failures, as seen in other studies. In contrast, failures occur primarily due to

fatigue stress in real oral condition. Therefore, further studies should be conducted to evaluate the variables effect analyzed in the present study. Additional clinical studies are needed to determine the long term prognosis of endodontically treated teeth restored using high-viscosity bulk fill resin composites.

Conclusion

Endodontically treated teeth restored using conventional nanohybrid resin composite present similar fracture resistance to different high-viscosity bulk fill composites.

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The authors deny any conflicts of interest.

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Tables

Table 1. Description of the composite resins used in the present study.

Composite Resin	Characteristics	Composition
Filtek^{MR} Z350 XT	Nano-hybrid composite resin Incremental technique Maximum increment thickness: 2 mm Cor: A2 Maker 3M ESPE, St. Paul, MN, USA	Silane treated ceramics, silane treated silica, UDMA, Bis-EMA (6), Bis-GMA, silane treated zirconia, polyethylene glycol dimethacrylate, triethylene glycol dimethacrylate, 2,6-di-tert-butyl-p-cresol. Load 78.5% weight, 59.5% vol
FiltekTM Bulk Fill	High viscosity Bulk Fill composite resin Maximum increment depth: 5 mm Color: A2 Maker 3M ESPE, St. Paul, MN, USA	Bis-GMA, Bis-EMA, EBPADMA, TEGDMA, procrylate-based resin, camphorquinone, EDMAB, Zirconia, silica, ytterbium trifluoride, silanized ceramics. Load 77% weight, 59% vol
SonicFillTM	High viscosity Bulk Fill composite resin Maximum increment depth: 5 mm Color: A2 Maker: Kerr, Orange, CA, USA	Bis-GMA, TEGDMA, EBPADMA, SiO ₂ , glasses and oxides Load 86% weight, 66% vol
Tetric[®] N-Ceram Bulk Fill	High viscosity bulk-fill composite resin Maximum increment depth: 4 mm Color: IVA Maker: Ivoclar Vivadent; Schaan, Liechtenstein	Bis-GMA, Bis-EMA, UDMA, barium glass, ytterbium trifluoride, mixture of oxides and prepolymers, additives, catalysts, stabilizers, pigments Load 78% weight, 55% vol (including prepolymer)

Bis-GMA, bisphenol A diglycidyl methacrylate; UDMA, Uretanodimethylmethacrylate; Bis-EMA (6), bisphenol A polyethylene glycol diether dimethacrylate; TEGDMA, triethyleneglycol dimethacrylate.

Table 2. Means and standard deviations (SDs) of fracture resistance of groups (n=10).

Groups	Mean $\pm$ SD. N^a
PCG Scotchbond™ Multi-Purpose + Filtek™ Z350XT	880.78 $\pm$ 287.09 ^a
NCG: No restoration.	215.14 $\pm$ 45.20 ^b
FBF Scotchbond™ Universal + Filtek™ Bulk Fill	759.56 $\pm$ 175.34 ^a
SF OptiBond™ All-in-one + SonicFill™	894.03 $\pm$ 231.30 ^a
TBF: Tetric®N- Bond Universal + Tetric®N-Ceram Bulk Fill	977.525 $\pm$ 336.03 ^a

Different letters indicate significant differences at level of significance $p < 0.05$.

Table 3. The frequency (%) of failure mode among the experimental groups (n=10).

Groups	<i>Favorable failures</i>	<i>Unfavorable failures</i>
PCG	80%	20%
NCG	30%	70%
FBF	70%	30%
SF	40%	60%
TBF	50%	50%

4 DISCUSSÃO

Na busca de um material restaurador com as melhores propriedades para restaurar dentes que foram tratados endodonticamente com moderada perda de estrutura coronária, o presente estudo procurou avaliar materiais restauradores com aplicação simplificada e comparar os mesmos com os sistemas já existentes considerados padrão-ouro.

Para restaurar os grupos experimentais (utilizando adesivos simplificados na técnica autocondicionante e resinas bulk-fill) o tempo necessário para concluir o procedimento restaurador era em média de 6 minutos, enquanto que quando os materiais tradicionais foram utilizados para restaurar as mesmas cavidades foram necessários em média 18 minutos. Praticidade e economia de tempo são características relevantes dentro da rotina de um consultório odontológico, no entanto existem mais propriedades desejáveis para que um material possa ser indicado para uso clínico.

No presente estudo avaliou-se a adaptação interna, a resistência de união e a resistência à fratura. Optou-se por utilizar resinas bulk-fill de alta viscosidade (condensáveis ou esculpíveis) pois, diferentemente das resinas bulk-fill flow, esses materiais podem ser aplicados em um único incremento, desde que a profundidade da cavidade permita, sem a necessidade de serem cobertos por uma resina composta convencional^{28,29}. Os sistemas adesivos foram aplicados na técnica autocondicionante, pois assim em um único passo clínico a dentina estaria hibridizada⁵⁵. As resinas e o adesivos que foram utilizados nesse estudo pertenciam a um mesmo fabricante²⁷.

O selamento da dentina foi feito imediatamente após a dentina ter sido tratada endodonticamente⁵⁶. O selamento imediato evita que materiais restauradores provisórios, como o cimento de óxido de zinco e eugenol e ionômero de vidro, o quais possuem propriedades tanto mecânicas como de selamento inferiores, acabem de alguma forma alterando o substrato dentinário que seria selado pelos sistemas adesivos e resinas bulk-fill. Clinicamente, dentes selados com esses materiais provisórios estão mais sujeitos tanto à fraturas do remanescente dental como a infiltração tanto coronária como radicular comprometendo assim a longevidade dos dentes que foram tratados endodonticamente^{2,3}.

A hipótese nula testada foi rejeitada uma vez que quanto à adaptação interna, a maior porcentagem de GAPs formou-se para a resina SonicFill e a menor para a resina Tetric N-Ceram Bulkfill. No período de 24 horas, não houve diferença estatisticamente significativa na resistência de união entre os sistemas adesivos estudados ($p>0,05$). No entanto, após o armazenamento de 1 ano, o Single Bond Universal, foi o único adesivo que não apresentou diferença estatisticamente significativa dos resultados encontrados após 24 horas ($p>0,05$). O Tetric N-Bond Universal apresentou os piores resultados de RU ($p<0,05$), os quais eram significativamente menores do que os valores de RU entrados após 24 horas. Quanto à avaliação da resistência à Fratura o grupo controle negativo (GCN, 215,14 N) apresentou os menores valores de resistência ($p<0,05$). Não foram encontradas diferenças estatisticamente significantes entre os demais grupos que estavam restaurados ($p>0,05$).

Um dos principais problemas das resinas compostas é a tensão de polimerização. Durante a reação de polimerização, ligações simples e duplas de monômeros de carbono irão formar cadeias poliméricas com ligações covalentes, isso resulta em uma contração volumétrica que varia de 1.5 a 5%. Existe ampla evidência científica de que a tensão resultante dessa reação de polimerização com consequente contração volumétrica pode levar a formação de fendas entre a cavidade e o material restaurador¹⁷. Os sistemas adesivos devem atuar como uma força contrária a essa tensão, no entanto quando a tensão de polimerização da resina composta excede a resistência de união entre os sistemas adesivo e a dentina, ocorre um descolamento do material restaurador das paredes da cavidade. Um agravante das cavidades endodônticas é que elas possuem um elevado fator C e a adaptação interna de restaurações com elevador fator C é menor quando comparada à cavidades com fator C menor⁵⁷.

Estudos já comprovaram que as substâncias utilizadas durante o tratamento endodôntico causam alterações no substrato dentinário, estas alterações resultam em uma diminuição da resistência de união³⁹⁻⁴². Nossos resultados mostraram que na análise imediata não houveram diferenças entre a resistência de união dos sistemas adesivos estudados. Mas quanto à formação de GAPs ocorreram diferenças. A maior formação de GAPs ocorreu no grupo restaurado com a resina SonicFill (31.56%), enquanto que a menor ocorreu no grupo restaurado pela Tetric N-Ceram Bulk-fill (12.32%).

Estudos anteriores já apresentaram bons resultados de adaptação para a Tetric N-Ceram Bulk-fill na dentina hígida^{27,57}, a composição do material poderia justificar esses resultados. No estudo Alqudaihi et al.⁵⁸, 2018 também foram encontrados os piores resultados quando a SonicFill foi utilizada. No estudo de Bennetti et al.⁵⁹, 2015 não houveram diferenças na contração de polimerização e na formação de GAPs entre as resinas SonicFill e Tetric N-Ceram Bulk-fill.

A formação de GAPs é um fenômeno complexo que depende da interação de muitos fatores. Nos estudos de adaptação envolvendo resinas bulk-fill todas as paredes da cavidade estavam localizadas em dentina^{27,57-59}. Não houveram diferenças entre a extensão dos GAPs formados entre a dentina e as resinas compostas utilizadas (que foram em média de 6,24µm). GAPs esses que foram muito menores do que os formados entre a dentina e o cimento de ionômero de vidro modificado por resina (que foram em média de 29,11 µm). Além dos GAPs formados entre a dentina e o cimento de ionômero de vidro modificado por resina serem maiores, na maioria das cavidades restauradas independente da resina utilizada, o CIVMR (Cimento de ionômero de vidro modificado por resina) se encontrava desadaptado da dentina. Já a adaptação entre a resina composta e o CIVMR foi perfeita, com exceção do SonicFill onde em metade das cavidades haviam GAPs entre o ionômero e a resina.

Sobre a Gutta-percha nos ensaios de adaptação interna e resistência à fratura foi aplicada uma fina camada de cimento de ionômero de vidro modificador por resina, esse procedimento é rotineiramente realizado clinicamente^{14,18,60}.

Em outros estudos realizados em cavidades profundas sem tratamento endodôntico, quando foi utilizada a “técnica sanduíche”, onde o ionômero ficou em contato com a dentina mais profunda e sobre ele foi aplicada a resina composta, os autores também encontraram GAPs maiores tanto de extensão como de largura do que os encontrados entre a dentina e a resina⁶⁰⁻⁶².

Como para a maioria das resinas o Ionoseal encontrava-se bem adaptado à resina acreditamos que quando a resina contraiu a resultante das forças acabou afetando a interface mais frágil, que no caso foi aquela encontrada entre o Ionoseal e a dentina levando assim às desadaptações entre esses materiais⁶⁰.

Os GAPs formados entre o Ionoseal e a Sonicfill podem ser causados por uma possível incompatibilidade química entre eles. No entanto inexistem estudos que avaliem a adaptação/ resistência de união entre esse materiais. Os resultados do

estudo de adaptação foram importantes e podem abrir margem para pesquisas futuras como resistência de união entre cimentos de ionômero de vidro modificados por resina e resinas bulk-fill, efeitos de se aplicar os sistemas adesivos e as resinas bulk-fill diretamente sobre a gutta-percha, os efeitos que o envelhecimento e a mastigação simulada poderiam causar sobre a adaptação, avaliar no mesmo espécime a adaptação e a resistência de união. Outros métodos de avaliação da adaptação também poderiam ser utilizados. Todas essas avaliações poderiam contribuir para encontrar as melhores estratégias de selar os dentes que foram endodonticamente tratados.

Atualmente os sistemas adesivos disponíveis podem ser classificados quanto à interação com o substrato dentinário. Assim existem os sistemas adesivos do tipo “condiciona e lava” e os sistemas adesivos autocondicionantes^{55,63}. Os sistemas do tipo condiciona e lava podem estar disponíveis em 3 (ácido fosfórico + primer + bond) ou em 2 passos (ácido fosfórico + primer e bond) operatórios. Já os sistemas autocondicionantes podem estar disponíveis em 2 (primer ácido + bond) ou em 1 (primer ácido e bond) passo operatório, as quais são também conhecidos como adesivos do tipo all-in-one^{63,64}.

Estudos anteriores já demonstraram que o uso de adesivos autocondicionantes na dentina pode ser benéfico para a longevidade adesiva^{55,65}. Já no esmalte, em virtude das características distintas desse substrato, o condicionamento ácido resulta em uma melhor efetividade adesiva^{63,66}. Com o objetivo de permitir o condicionamento com ácido fosfórico no esmalte e o uso de monômeros autocondicionantes na dentina utilizando um único frasco de adesivo, alguns fabricantes introduziram no mercado os chamados “Adesivos Universais”. Esses adesivos fornecem ao clínico a versatilidade de utilizar ambas estratégias, ou seja, tanto condiciona e lava como a autocondicionante⁶⁷.

Sendo assim os adesivos mais simplificados existentes no mercado hoje são: os sistemas autocondicionantes de 1 passo operatório e os sistemas adesivos universais utilizados na técnica autocondicionante. Dos sistemas adesivos utilizados no presente estudo o Scotchbond Multi-Purpose é um sistema adesivo condiciona e lava de 3 passos, o Single Bond Universal e o Tetric N-Bond Universal são sistemas adesivos universais, os quais foram empregados na técnica autocondicionante e por fim o Optibond All-in-one é um adesivo autocondicionante de 1 passo.

Associado ao fato de que quanto à resistência de união não houveram diferenças na análise imediata entre os sistemas adesivos estudados, na análise de microspécia confocal imediata todos os adesivos apresentaram uma boa penetração à dentina. Sabe-se que a retenção micromecânica é essencial para a resistência de união imediata. Esta boa penetração era esperada para o adesivo Scotchbond Multipurpose, pois neste sistema é feito o condicionamento ácido o qual promove uma completa remoção da smear layer^{55,63}. No entanto na dentina normal, a interação que ocorre entre os adesivos autocondicionantes e a dentina é mais superficial, diferindo do que foi observado através das imagens de microscopia confocal.

Nos adesivos autocondicionantes a agressividade, depende do pH e pode alterar a profundidade de interação entre o substrato dental e estes adesivos⁶⁸. De acordo com os fabricantes, o pH dos adesivos utilizados variou de 2,5 à 3, sendo assim esses adesivos podem ser classificados como ultra-leves. Adesivos ultra leves, tendem a formar uma interface adesiva fina e sub-micrométrica, onde os resin tags são pouco frequentes⁶⁴. Os adesivos estudados mesmo com pH mais fraco podem ter apresentado uma melhor penetração na dentina tratada endodonticamente devido ao aumento na permeabilidade causado pelo NaOCl⁶⁹ e pelo EDTA⁷⁰, estas substâncias provavelmente criaram uma smear layer que difere daquela encontrada na dentina sem tratamento endodôntico.

Estudos anteriores que avaliaram a resistência de união entre os sistemas adesivos e a dentina tratada endodonticamente^{56,73-75}, concluíram que o tratamento endodôntico foi capaz de reduzir a resistência de união à dentina. Segundo esses estudos o principal responsável pela diminuição da resistência de união foi o NaOCl uma vez que esse irrigante interfere na polimerização dos materiais resinosos⁷¹. Radicais livres remanescentes do NaOCl na dentina competem com a propagação de radicais vinílicos gerados durante a fotoativação dos sistemas adesivos, isso resulta em uma terminação prematura de cadeia e incompleta polimerização^{72,73}. Outros estudos também mostraram que o cimento endodôntico e o EDTA são capazes de contribuir para a redução da resistência de união.

Nossos resultados após um ano, mostraram que os sistemas adesivos simplificados estudados apresentaram comportamentos distintos, uma vez que para alguns adesivos ocorreu a diminuição da resistência de união enquanto que para outros não. Acreditamos que apesar do tratamento endodôntico poder interferir nos

resultados imediatos, a longevidade adesiva é influenciada principalmente pela composição dos materiais.

Durante a aplicação sistema adesivo de 3 passos “etch-and-rinse” Scotchbond Multi Purpose é importante realizar a manutenção da umidade para evitar o colabamento das fibrilas colágenas⁶³, ainda devido ao gradiente de concentração, algumas regiões que foram desmineralizadas não são completamente preenchidas pelo sistema adesivo, isso pode formar defeitos no interior da camada híbrida⁷⁶. Nessas condições, enzimas metaloproteínases (MMPs) endógenas presentes na matrix colágena podem levar a uma degradação da interface adesiva, a qual afeta a eficácia ao longo do tempo dos sistemas adesivos⁷⁸. Assim como ocorre na dentina normal⁷⁷⁻⁷⁹, a dentina endodonticamente tratada também foi vulnerável a degradação hidrolítica após 1 ano de armazenamento. Uma vez que este adesivo apresentou redução da RU. As imagens de microscopia confocal podem ajudar a ilustrar esse fenômeno.

O Single Bond Universal, foi o único adesivo que não apresentou redução da resistência de união após 1 ano de armazenamento. Diferentemente do Scotchbond Multi-Purpose e do Optibond All-in-one nas imagens de microscopia confocal, não foi encontrado água na camada híbrida após o envelhecimento. Na composição deste adesivo inclui o monômero funcional 10 MDP. Comparado a outros monômeros funcionais este apresenta uma longa cadeia espaçadora, a qual é capaz de criar uma união intensa e estável, essa cadeia tem mostrado contribuir na proteção da camada híbrida contra a degradação, resultando assim em uma melhor durabilidade adesiva^{80,81}. Na dentina normal o resultado de outros estudos corroboram com os nossos resultados⁸².

O monômero funcional do sistema adesivo Optbond All-in-one é o glycerol-fosfato dimetacrilato (GPDM). Um recente estudo concluiu que a ligação formada entre esse monômero e a hidroxiapatita é mais fraca quando comparado com a forte união que existe entre o MDP e a hidroxiapatita. Assim por não formar de um sal de cálcio estável, adesivos contendo esse monômero pode apresentar uma menor resistência de união⁸¹. Segundo nossos resultados, comparados aos resultados iniciais este adesivo apresentou uma redução da resistência de união após o envelhecimento. As imagens de microscopia confocal revelaram a presença de água no interior da camada híbrida. No entanto, mesmo que a resistência de união tenha

diminuído após o envelhecimento, não houveram diferenças entre este adesivo e o Scotchbond Multi Purpose e o Single Bond Universal após 1 ano.

A mistura e diluição do 10-MDP com outros monômeros para produzir novos adesivos por diferentes fabricantes, assim como as diferentes concentrações que podem existir desse monômero nos diferentes adesivos, podem impactar na habilidade do 10MDP em estabelecer uma união estável⁸². No sistema adesivo Tetric N-Bond Universal além do monômero 10 MDP também há o monômero funcional MCAP, o qual também é capaz de reagir e estabelecer união com a hidróxiapatita. Esse fato pode ajudar a explicar os piores valores de resistência de união encontrados para este adesivo após ano, uma vez que o 10 MDP e o MCAP podem ter competido pelo cálcio existente na dentina. Além disso, a pureza do 10 MDP sintetizada por diferentes fabricantes pode resultar em diferentes comportamentos adesivos⁸¹. Ainda os piores valores de RU pode estar associados aos tags fraturados encontrados nas imagens de microscopia confocal.

Por último foi feita a análise da resistência à fratura de dentes tratados endodonticamente restaurados com adesivos simplificados e resinas bulk-fill. A maioria dos estudos que empregaram resinas bulk-fill em dentes tratados endodonticamente avaliaram a resistência à fratura^{9,10,18,38,54}, no entanto, o nosso estudo foi o primeiro a empregar exclusivamente resinas de alta viscosidade.

Como já foi mencionado anteriormente os compósitos bulk-fill estão disponíveis em alta e baixa viscosidade. Para diminuir a viscosidade dos materiais os fabricantes reduzem a % de partículas de carga, e isso tem consequências nas propriedades mecânicas dos mesmos¹⁸. Assim os fabricantes recomendam que os compósitos do tipo flow sejam recobertos por uma resina convencional, a qual apresenta melhores propriedades mecânicas. Por outro lado, os materiais de alta viscosidade, apresentam um alto conteúdo de carga, melhores propriedades mecânicas e dependendo da profundidade da cavidade permite que o material seja aplicado em um único incremento^{29,83}. Como buscávamos as estratégias mais simplificadas para selar os dentes que foram endodonticamente tratados esse último foi o material selecionado.

A utilização dos sistemas simplificados não apresentou diferenças quanto à resistência à fratura quando comparados aos dentes que foram restaurados com um adesivo condiciona e lava de 3 passos e uma resina composta nanoparticulada na técnica incremental. Esses resultados podem ser uma consequência do alto conteúdo de carga que todas as resinas empregadas nesse estudo apresentavam (77 a 86%

em peso). Por outro lado, como esperado, os piores resultados de resistência à fratura ocorreram para os dentes tratados endodonticamente sem restaurações.

A qualidade do selamento definitivo dos dentes que foram tratados endodonticamente é fundamental para o prognóstico do mesmo. No entanto, muitos fatores podem estar envolvidos, como as alterações causadas no substrato pelos irrigantes e cimentos, a utilização de selamentos provisórios previamente à restauração definitiva, a utilização de cimentos de ionômero de vidro sobre a gutta percha.

Nosso estudo procurou focar nas estratégias simplificadas para realizar o selamento desses dentes. Com as devidas limitações, de acordo com os nossos resultados, podemos sugerir que, mesmo que o tratamento endodôntico prejudique a adesão inicial é vantajoso a utilização do Single Bond Universal e Optibond All-in-one pois além de serem adesivos simplificados eles não apresentaram resultados diferentes dos sistemas condiciona e lava de três passos após 1 ano. O Single Bond Universal ainda manteve, graças à sua composição, os valores de adesão iniciais. Quanto a adaptação interna das restaurações, o cimento de ionômero de vidro modificado por resina, pode ter causado mudanças na resultante das forças, uma vez que ele se encontrava mais desadaptado e com fendas maiores do que as resinas utilizadas. Já quanto à adaptação das resinas, a Tetric N-Ceram Bulk-fill apresentou os melhores resultados e a Sonicfill os piores. Por último não houve diferenças entre as resinas utilizadas quanto à resistência à fratura. A partir dos nossos achados mais estudos deveriam continuar investigando a respeito das melhores estratégias para se restaurar definitivamente a dentina que foi endodonticamente tratada.

5 CONCLUSÃO

Quanto à adaptação interna avaliada através da microscopia eletrônica de varredura, a maior porcentagem de GAPs formou-se para a resina SonicFill, e a menor para a resina Tetric N-Ceram Bulk-fill.

A utilização de sistemas adesivos simplificados na dentina tratada endodonticamente produziu resultados imediatos de resistência de união, ao ensaio de microtração, similares ao sistema adesivo de 3 passos convencional.

Depois de um ano, o Single Bond Universal manteve os valores de resistência de união. Os sistemas adesivos simplificados Single Bond Universal e Optibond All-in-one apresentaram resultados de resistência de união similares ao Scotchbond Multi Purpose. O adesivo simplificado Tetric N-Bond Universal, apresentou redução da resistência de união inicial e os piores resultados de RU após o envelhecimento.

Os dentes tratados endodonticamente sem restauração apresentaram os piores resultados quanto à resistência à fratura. Dentes tratados endodonticamente restaurados com resina composta nano-híbrida apresentaram resistência à fratura similar aos restaurados com resinas bulk-fill de alta viscosidade avaliados.

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¹ De acordo com o Guia de Trabalhos Acadêmicos da FOAr, adaptado das Normas Vancouver. Disponível no site da Biblioteca: <http://www.foar.unesp.br/Home/Biblioteca/guia-de-normalizacao-atualizado.pdf>

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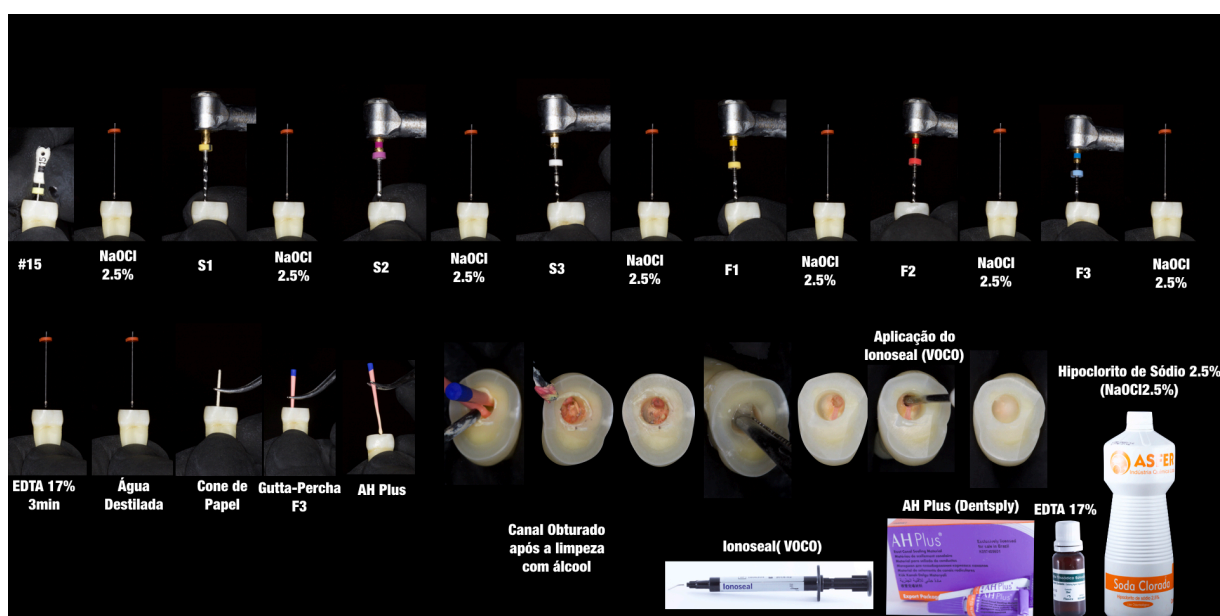
APÊNDICE A – Materiais e Métodos

Figura A1 - Sistemas restauradores avaliados no presente estudo.



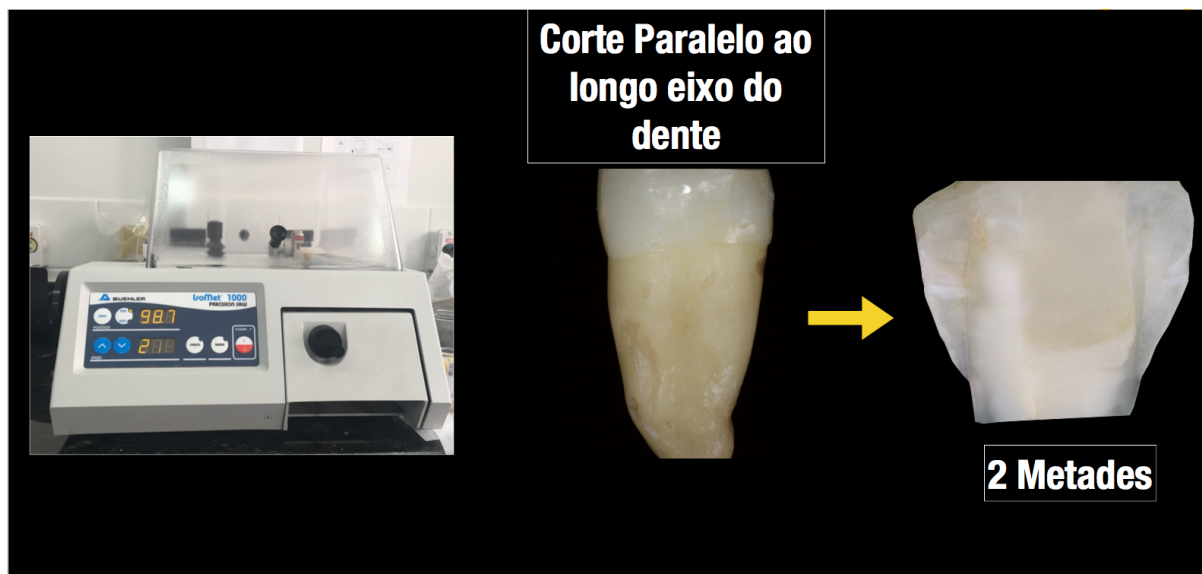
Fonte: Elaboração própria

Figura A2 - Tratamento endodôntico dos espécimes, limpeza da cavidade e aplicação do cimento de ionômero de vidro modificado por resina. Protocolo utilizado no teste de adaptação interna e resistência de união.



Fonte: Elaboração própria

Figura A3 - Após as restaurações serem concluídas, obtenção das duas metades das restaurações, as quais foram avaliadas no ensaio de adaptação interna



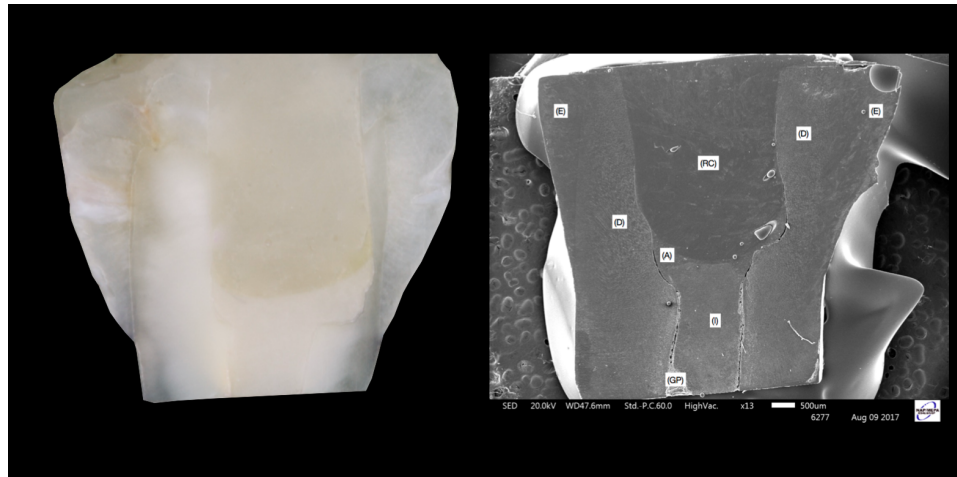
Fonte: Elaboração própria

Figura A4 - Confeccção das réplicas em resina epóxi, das amostras que foram avaliadas no ensaio de adaptação interna.



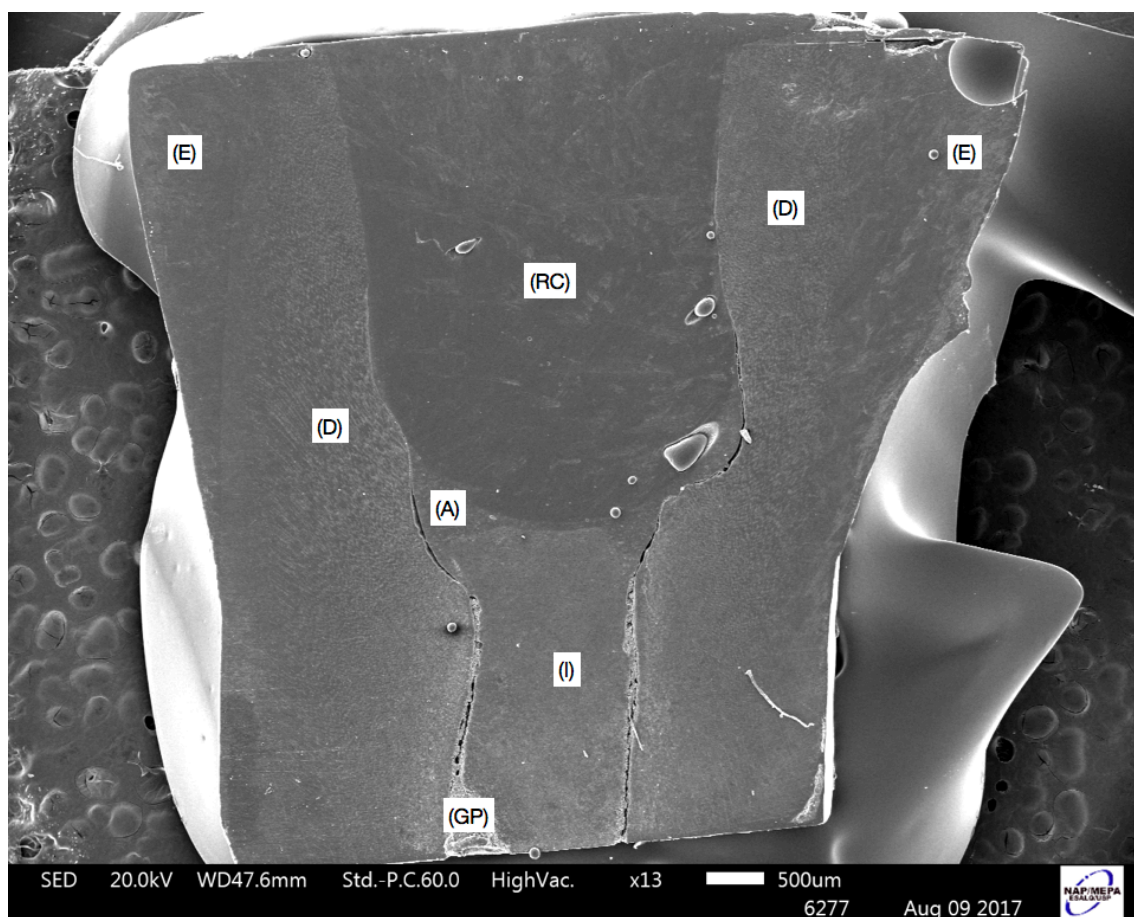
Fonte: Elaboração própria

Figura A5 - Imagens formadas à partir dos espécimes.



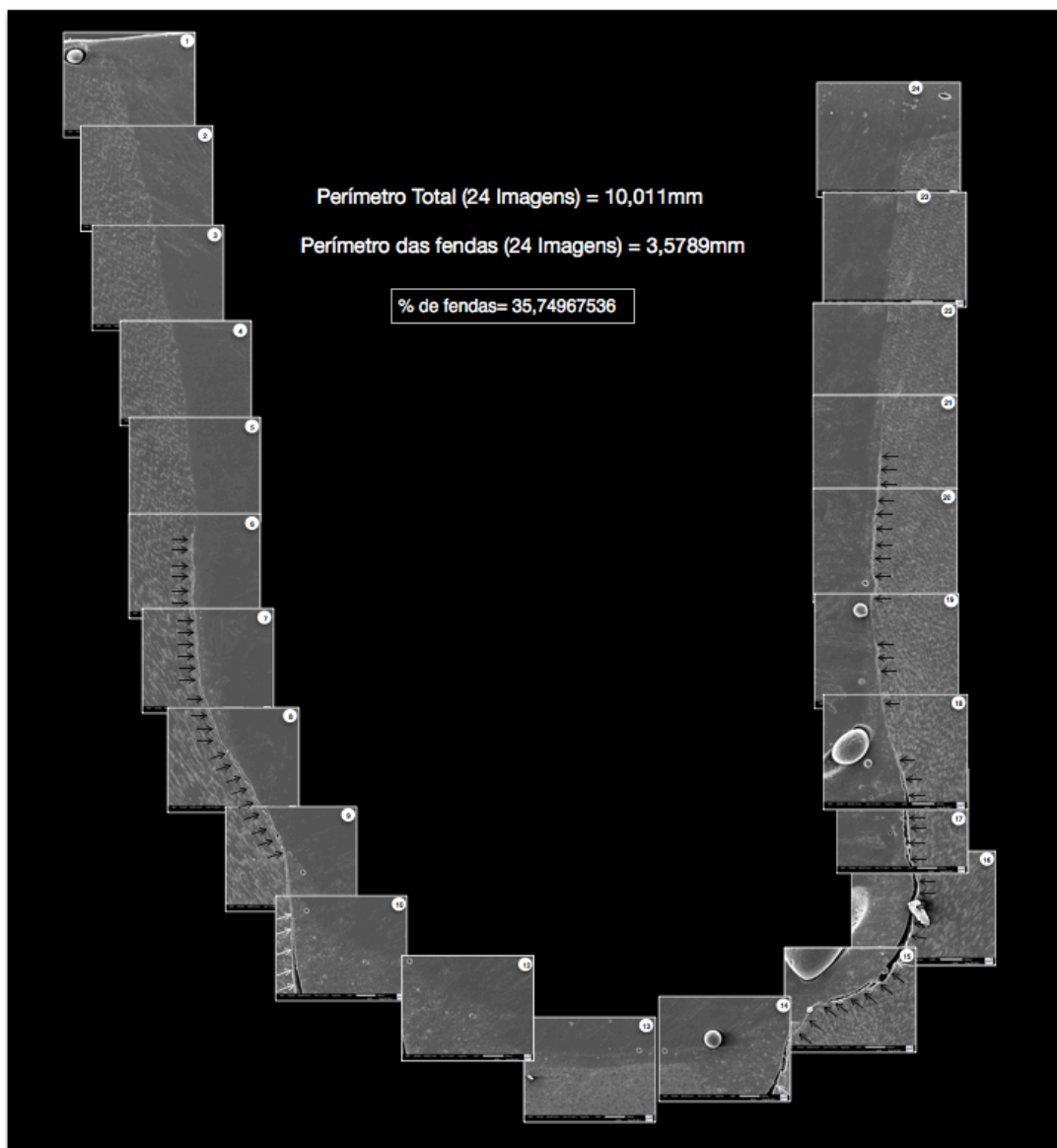
Fonte: Elaboração própria

Figura A6 - Microscopia eletrônica de varredura da adaptação marginal interna (dente cortado longitudinalmente) com aumento de 13x, para reconstruir essa interface resina/dentina e analisar melhor as desadaptações foram necessárias 24 imagens com aumento de 200x. **(E)** Esmalte; **(D)** Dentina; **(RC)** Resina Composta; **(A)** Adesivo; **(I)** Ionomêro de vidro modificado por resina – Ionoseal; **(GP)** Gutta-Percha. Nota-se que as fendas entre o Ionoseal são visíveis mesmo nesse aumento menor, e são maiores do que as encontradas entre a resina composta e a dentina.



Fonte: Elaboração própria

Figura A7- 24 Imagens com aumento de 200x para reconstruir toda a interface proximal da restauração. Setas pretas indicam desadaptação entre a resina e a dentina. Setas brancas indicam desadaptação do lonoseal e a dentina. Medida dos perímetro total das 24 imagens, assim como da somatória das fendas encontradas entre a resina e o dente e a resina e o lonoseal. % das fendas formadas para esta restauração. Neste exemplo o valor que seria utilizado para o cálculo da média séria 35,74%.



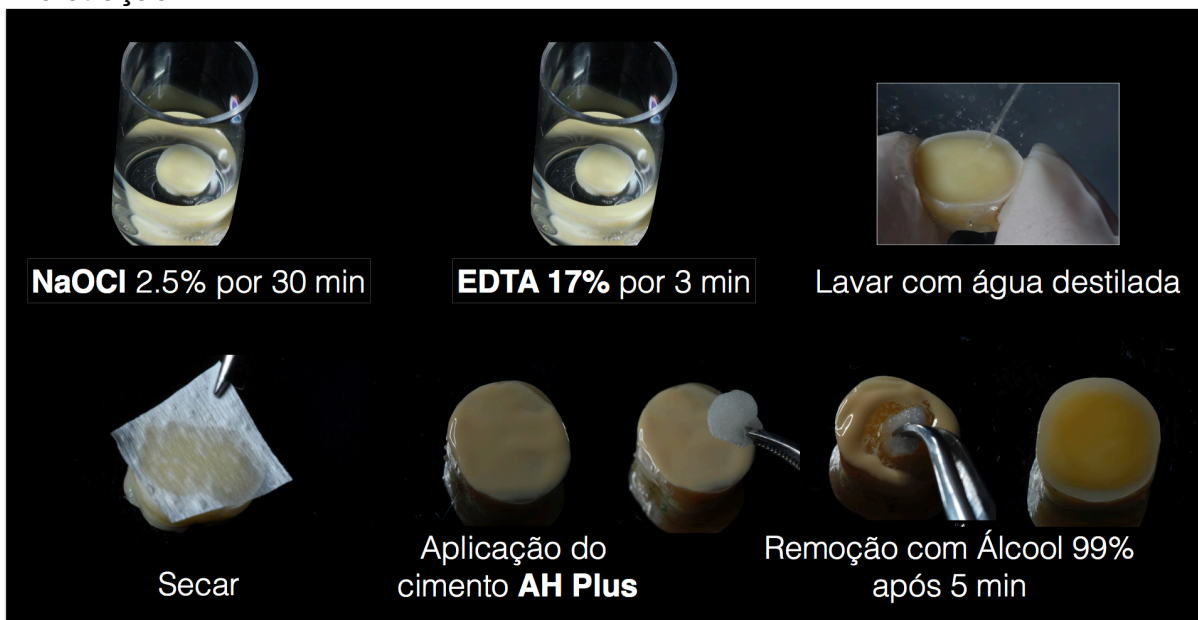
Fonte: Elaboração própria

Figura A8 - Remoção da superfície oclusal.



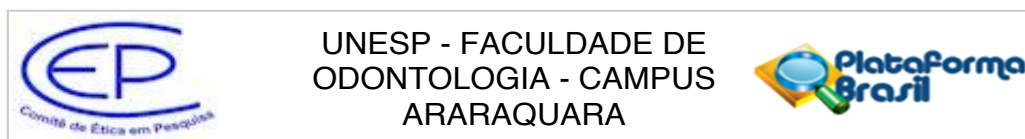
Fonte: Elaboração própria

Figura A9 - Simulação do tratamento endodôntico, previamente ao ensaio de microtração.



Fonte: Elaboração própria

ANEXO A – Comprovante Comitê de Ética e Pesquisa Faculdade de Odontologia de Araraquara.



COMPROVANTE DE ENVIO DO PROJETO

DADOS DO PROJETO DE PESQUISA

Título da Pesquisa: Avaliação da adaptação marginal interna, resistência de união e resistência à fratura da associação entre: Resinas Bulk-fill e adesivos universais em dentes tratados endodonticamente

Pesquisador: Milton Carlos Kuga

Versão: 1

CAAE: 68339217.2.0000.5416

Instituição Proponente: Faculdade de Odontologia de Araraquara - UNESP

DADOS DO COMPROVANTE

Número do Comprovante: 048858/2017

Patrocinador Principal: FUND COORD DE APERFEICOAMENTO DE PESSOAL DE NIVEL

Informamos que o projeto Avaliação da adaptação marginal interna, resistência de união e resistência à fratura da associação entre: Resinas Bulk-fill e adesivos universais em dentes tratados endodonticamente que tem como pesquisador responsável Milton Carlos Kuga, foi recebido para análise ética no CEP UNESP - Faculdade de Odontologia - Campus Araraquara em 15/05/2017 às 14:59.

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**Não autorizo a publicação deste trabalho pelo prazo de 2 anos após a data de
defesa.**

[Direitos de publicação reservado ao autor]

Araraquara 21 de março de 2019

Aline Carvalho Giroto