UNIVERSIDADE ESTADUAL DE CAMPINAS
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MARINA RODRIGUES SANTI

AVALIAÇÃO DE TRATAMENTOS DENTINÁRIOS INTRA-RADICULARES ALTERNATIVOS NA RESISTÊNCIA DE UNIÃO DE CIMENTOS RESINOSOS AUTO-ADESIVOS

EVALUATION OF ALTERNATIVE PRETREATMENTS ON INTRA-RADICULAR DENTIN BOND STRENGTH OF SELF-ADHESIVE RESIN CEMENTS

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Dissertação apresentada à Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas como parte dos requisitos exigidos para a obtenção do título de Mestra em Clínica Odontológica na Área de Dentística.

Dissertation presented to the Piracicaba Dental School of the University of Campinas in partial fulfillment of the requirements for the degree of Master in Clinical Dentistry in Operative Dentistry area.

Orientador: Prof. Dr. Luís Roberto Marcondes Martins

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RESUMO

Objetivo(s): O objetivo deste estudo foi avaliar a influência de pré-tratamentos alternativos na resistência de união de cimentos resinosos auto-adesivos (SARCs) no canal radicular após a cimentação de pinos de fibra de vidro. **Materiais e Métodos:** Dois SARCs foram utilizados: Maxcem Elite (MAX – Kerr - Orange, CA, EUA) e Calibra Universal (CAL - Dentsply Sirona - York, PA, EUA); e dois pré-tratamentos dentinários foram aplicados: 26% de ácido poliacrílico (PA) por 10s, e 2,5% de tetrafluoreto de titânio (TiF_4) por 60s. Os grupos que não receberam os pré-tratamentos foram considerados como controle. Sessenta incisivos bovinos foram divididos aleatoriamente em 6 grupos (n=10) e após sete dias da cimentação dos pinos de fibra de vidro, as amostras foram submetidas ao teste de resistência de união por push-out (POBS). O modo de falha, a morfologia da superfície (n=3) e a análise da interface adesiva (n=3) foram observadas através de microscopia eletrônica de varredura (SEM). A porcentagem de infiltração dos SARCs (n=3) foi analisada na interface adesiva com magnificação de 63x, e em 20x de acordo com os terços do canal radicular (cervical, médio e apical), através da microscopia de varredura confocal a laser (CLSM). As imagens foram encaminhadas para o software *Image J*. Os dados foram analisados através de ANOVA três fatores com teste *post-hoc* de Bonferroni (POBS e CLSM) (α=5%). O padrão de fratura, morfologia de superfície e interface adesiva foram analisados de modo descritivo. **Resultados:** O cimento MAX apresentou aumento nos valores de push-out no terço cervical quando ambos os pré-tratamentos foram aplicados, e no terço médio com TiF_4. O cimento CAL apresentou maiores valores no terço cervical somente quando o PA foi aplicado. Falhas do tipo I (falha entre a dentina e o cimento resinoso) foram as mais predominantes no grupo controle e para os grupos pré-tratados com PA. Para os grupos que receberam o TiF_4, a falha mais predominante foi a do tipo V (falha mista). A morfologia de superfície apresentou túbulos dentinarios amplos quando o PA foi aplicado e formação de aglomerados quando utilizado o TiF_4. As imagens de interface de SEM e CLSM revelaram uma maior quantidade de tags resinosos para ambos os pré-tratamentos. As imagens de 20x de CLSM apresentaram maior homogeneidade na penetração dos cimentos quando associados aos pré-tratamentos. **Conclusão:** O tratamento para dentina intra-radicular com PA e TiF_4, aumentou os valores de POBS independente do cimento utilizado, e foi capaz de formar uma maior quantidade de tags resinosos comparado ao grupo controle dependendo da composição do SARC utilizado.

Palavras-chave: Pino de fibra de vidro; Cimentos resinosos; Dentina.

ABSTRACT

Objectives: The objective of this study was to evaluate the influence of pretreatments on intra-radicular bond strength of self-adhesive resin cements (SARCs) after fiber glass post cementation. **Materials and Methods:** Two SARCs were used: Maxcem Elite (MAX– Kerr - Orange, CA, USA) and Calibra Universal (CAL- Dentsply Sirona - York, PA, USA); and two pretreatments were applied: 26% polyacrylic acid (PA) for 10s and 2.5% titanium tetrafluoride (TiF_4) for 60s. The two remaining groups that did not received a pretreatment were considered as a control. Sixty bovine incisors were randomly divided into 6 groups (n=10) and after seven days of the cementation of the fiber glass post, the samples were subjected to the push-out bond strength test (POBS). Failure mode, surface morphology (n=3) and adhesive interface analysis (n=3) were observed by a Scanning Electron Microscopy (SEM). The resin cement infiltration (n=3) was analyzed on the adhesive interface in 63x magnification and in 20x according to radicular root thirds (cervical, medium, and apical) by confocal laser scanning microscopy (CLSM). The imagens were forwarded to the Image J software. Data were analyzed by three-way ANOVA and Bonferroni *post-hoc* test (POBS and CLSM) (α=5%). The failure mode, surface morphology and adhesive interface were analyzed descriptively. **Results:** MAX presented an increase POBS values in the cervical third for both pretreatments, and in the middle third for TiF_4. CAL presented higher values in the cervical third only when PA was applied. Failure type-I (between the dentine and the resin cement) was the most prevalent failure mode for control and PA groups, and TiF_4 was mixed failures (type-V). The surface morphology presented openned dentin tubules for PA and agglomerates for TiF_4. The SEM and CLSM images reveled a large quantity resin tags for both pretreatments. The 20x imagens by CLSM presented higher homogeneity penetration of SARCs when the pretreatments were applied. **Conclusion:** Dentin pretreatment with PA and TiF_4 solutions improved the POBS regardless the resin cement, and it was able to form more resin tags compared to control groups depending on the SARC composition.

Key Words: Fiberglass post; Resin cements; Dentin.

SUMÁRIO

1 INTRODUÇÃO

Atualmente, fatores estéticos associados aos conceitos de preservação da estrutura dental, têm contribuído para o aprimoramento de técnicas restauradoras reconstrutivas (Abo-Hamar et al., 2005). Exemplo disso, é a reabilitação de dentes tratados endodonticamente com grande perda de estrutura coronal que requer, em alguns casos, o uso de retentores intrarradiculares (Machado et al., 2015; Webber et al., 2015).

Dentre os retentores intrarradiculares, o pino de fibra de vidro possui um módulo de elasticidade semelhante ao da dentina e aos materiais resinosos, proporcionando distribuição homogênea das cargas oclusais, o que diminui o risco de fratura radicular (Bosso et al., 2015; Maroulakos et al., 2015). Porém, existem vários desafios em relação ao procedimento de cimentação deste material, visto que os canais radiculares possuem configuração cavitária desfavorável e baixo alcance de luz nas regiões mais apicais da raiz (Faria-e-Silva et al., 2010). Sendo assim, cimentos resinosos duais são os mais indicados nesta situação (Aguiar et al., 2010).

Os cimentos resinosos duais são ativados quimicamente e através da fotoativação, visando garantir que a polimerização ocorra mesmo na ausência de luz (Aguiar et al., 2010). Além disso, estes cimentos apresentam melhores propriedades mecânicas e baixa sorção e solubilidade quando comparados aos cimentos odontológicos tradicionais como fosfato de zinco e ionômero de vidro (Tanoue et al., 2003).

Mas apesar das vantagens, os cimentos resinosos duais apresentam uma técnica de aplicação complexa que pode comprometer a qualidade da adesão devido aos vários passos operatórios a serem seguidos, podendo ocasionar o insucesso da cimentação dos pinos de fibra de vidro (Yang et al., 2006). Esta categoria de cimentos exige a aplicação de sistema adesivo prévio à cimentação, porém, existe uma incompatibilidade entre diferentes marcas de cimentos e adesivos simplificados, principalmente adesivos autocondicionantes de passo único (Cheong et al., 2003). Segundo autores, essa incompatibilidade deve-se à presença de monômeros resinosos ácidos residuais na camada adesiva não polimerizada pela inibição do oxigênio e que reagem com a amina terciária da resina composta. Com isso, a amina é neutralizada não podendo assim reduzir o peróxido de benzoíla na reação redox, responsável pela polimerização do compósito (Cheong et al., 2003; Tay et al., 2003).

Sendo assim, essa incompatibilidade leva a ausência da completa polimerização, criando uma área susceptível à propagação de fraturas que resulta em menores valores de resistência de união (Faria-e-Silva et al., 2010). Portanto, com o objetivo de diminuir os passos clínicos operatórios necessários e compensar as limitações encontradas nas técnicas atualmente empregadas, surgiram os cimentos resinosos autoadesivos (Aguiar et al., 2010).

Os cimentos resinosos autoadesivos (SARCs) foram desenvolvidos para simplificar o procedimento de cimentação devido ao fato de não exigirem as etapas de condicionamento ácido e adesivo prévio (Ferracane et al., 2011; Hitz et al., 2012; Farina et al., 2016; Manso e Carvalho, 2017). No entanto, ao comparar essa categoria de cimentos com cimentos resinosos duais, tem sido relatado na literatura, problemas associados às propriedades mecânicas, como menores valores de resistência de união (Hitz et al., 2012; Miotti et al., 2020). E este fato pode estar relacionado ao mecanismo de adesão dos SARCs, por possuírem monômeros ácidos na composição (Ferracane et al., 2011; Zorzin et al., 2012; Madruga et al., 2013; Manso; Carvalho, 2017).

Os monômeros ácidos são predominantemente monômeros de acrilato ou metacrilato, com grupos de ácido carboxílico ou fosfórico, e a concentração destes monômeros é de extrema importância durante o procedimento de cimentação, pois é preciso uma quantidade alta o suficiente para garantir a desmineralização e ao mesmo tempo, uma quantidade baixa para evitar hidrofiliação excessiva e comprometer a estabilidade mecânica por absorção de água (Ferracane et al., 2011; Hitz et al., 2012; Manso; Carvalho, 2017). Desta forma, é necessário que haja um equilíbrio para que a adesão mecânica ocorra quando os monômeros ácidos ao se ligarem ao cálcio da hidroxiapatita na camada desmineralizada de smear layer, criem uma ligação através de ácido fosfórico ionizado (Madruga et al., 2013).

No entanto, a camada de smear layer criada durante a preparação do conduto radicular para cimentação de um pino de fibra de vidro, também pode estar relacionada a baixas propriedades mecânicas de adesão dos SARCs (Faria-e-Silva et al., 2013; Jitumori et al., 2019). A camada de semar layer produzida no canal radicular é mais espessa e densa do que a observada na dentina coronal, e a presença desta camada prejudica um contato adequado entre os SARCs e a dentina subjacente, interferindo na força de união entre cimento e dentina (Faria-e-Silva et al., 2013; Jitumori et al., 2019). Assim, a escolha de soluções ácidas para remover parcialmente ou modificar a smear layer superficial poderia aumentar a adesão e melhorar as propriedades mecânicas dos SARCs (Monticelli et al., 2008; Faria-e-Silva et al., 2013).

Portanto, devido à similaridade dos SARCs com o mecanismo de adesão dos ionômeros de vidro, o ácido poliacrílico tem sido recomendado como um pré-tratamento dentinário alternativo (Monticelli et al., 2008; Pavan et al., 2010). O ácido poliacrílico é um ácido categorizado como leve (pH=2), que contém numerosos grupos de íons carboxílicos, que podem formar uma variedade de ligações de hidrogênio e remover parcialmente a smear layer, ocasionando uma maior interação entre o cimento e a dentina. Pavan e colaboradores., (2010) observaram que o ácido poliacrílico melhorou a resistência à microtração e pode ser incorporado como uma etapa de limpeza em procedimentos de cimentação. Mas, apesar do impacto positivo no desempenho do ácido poliacrílico, existem resultados conflitantes na literatura (Faria-e-Silva et al., 2013).

Outro composto que vem sendo estudado é o tetrafluoreto de titânio (TiF_4). Este composto fluoretado é classificado como um ácido intermediário forte (pH = 1,5) que possui forte ligação entre o titânio e o átomo de oxigênio do grupo fosfato, formando assim uma camada vítrea resistente a ácidos e soluções alcalinas (Wahengbam et al., 2011; Tranquilin et al., 2016; Torres et al., 2017; Viana et al., 2019). Como os SARCs possuem monômeros ácidos com cadeia fosfórica na composição, a incorporação de TiF_4 na camada híbrida pode desempenhar um papel importante durante o procedimento de adesão, mas essa hipótese nunca foi estudada na literatura.

Dündar et al., (2011), propuseram a aplicação de uma solução aquosa de TiF_4 por 60s na dentina antes da aplicação de adesivos que contenham monômeros ácidos. Basting e colaboradores (2014), avaliaram a dentina tratada com tetrafluoreto de titânio 2,5%, e verificaram que este composto pode modificar a micromorfologia da superfície dentinária e formar uma camada resistente ao ácido que não pode ser removida, mesmo após o uso de ácido cítrico por 30 mim. Além disso, Tranquilin et al., (2016) observaram que uma camada híbrida com a formação de numerosos tags resinosos foi obtida, mostrando os efeitos favoráveis do uso de TiF_4 2,5% como pré-tratamento antes da aplicação de um sistema adesivo autocondicionante de dois passos.

Portanto, uma simples etapa prévia ao procedimento de cimentação poderia contribuir para melhorar as propriedades mecânicas, dessa forma, o objetivo deste estudo *in vitro* foi avaliar a influência de pré-tratamentos na dentina radicular na resistência de união de cimentos resinosos autoadesivos utilizados para cimentação de pinos de fibra de vidro, além do modo de fratura, morfologia de superfície e interface adesiva. As hipóteses nulas foram: (1) a força de união de ambos os SARCs não é influenciada pela técnica de pré-tratamento aplicada entre os diferentes terços da raiz da dentina radicular; (2) o modo de falha não é influenciado pelos pré-tratamentos; e (3) a camada híbrida com formação de tags resinosos não é influenciada pelo tipo de pré-tratamento aplicado.

2 ARTIGO: Evaluation of alternative pretreatments on intra-radicular dentin bond strength of self-adhesive resin cements.

Artigo submetido ao periódico Clinical Oral Investigation (Anexo 2).

Marina Rodrigues Santi; Rodrigo Barros Esteves Lins; Beatriz Ometto Sahadi; Jorge Rodrigo Soto-Monteiro; Luís Roberto Marcondes Martins

Abstract

Objectives: The objective of this study was to evaluate the influence of pretreatments on dentin bond strength of self-adhesive resin cements (SARCs) on fiber glass post cementation. **Materials and Methods:** Two SARCs were used: Maxcem Elite (MAX) and Calibra Universal (CAL); and two pretreatments: 26% polyacrylic acid (PA) and 2.5% titanium tetrafluoride (TiF_4). The groups that did not received a pretreatment were considered as a control. Sixty bovine incisors were randomly divided into 6 groups (n=10) and subjected to the push-out bond strength test (POBS). Failure mode, surface morphology (n=3) and adhesive interface analysis (n=3) were observed by a Scanning Electron Microscopy (SEM). The resin cement infiltration (n=3) was investigated by confocal laser scanning microscopy (CLSM). Data were analyzed by three-way ANOVA and Bonferroni *post-hoc* test with a significance level set at 5% (POBS and CLSM). The failure mode, surface morphology and adhesive interface were analyzed descriptively. **Results:** MAX presented an increase POBS values in the cervical third for both pretreatments, and in the middle third for TiF_4. CAL presented higher values in the cervical third only when PA was applied. Failure type-I (between the dentine and the resin cement) was the most prevalent failure mode for control and PA groups, and TiF_4 was mixed failures (type-V). The surface morphology presented openned dentin tubules for PA and agglomerates for TiF_4. The SEM and CLSM images reveled a large quantity resin tags for both pretreatments. **Conclusion:** Dentin pretreatment with PA and TiF_4 solutions improved the POBS and depending on the SARC composition, it was able to improve the resin tag formation.

Key Words: Fiberglass; Resin cements; Hybrid layer; Dentin; Titanium tetrafluoride

1. Introduction

Self-adhesive resin cements (SARCs) were developed to simplify the adhesive luting procedure because they do not require the etching, priming and bonding steps, according to the manufacturers. Due to the difficult to access canals with adhesives, this category of resin cement has been recommended to fiber glass post cementation. [1-4] However, when comparing with conventional resin cements, it has been reported problems associated to mechanical properties, such as lower values of bond strength, that may be related to the mechanism of adhesion of the acidic monomers present in the composition. [1-7]

Therefore, the concentration of the acidic monomers is extremely important during the luting procedure because it needs to be high enough to guarantee proper demineralization of the smear layer and bonding to dentin and enamel, and as low as possible to avoid excessive hydrophilicity and compromise mechanical stability by excessive absorption of water. [1-3] However, the smear layer created during the root preparation is thicker and denser than the observed in coronal dentin, and the presence of this layer impairs a proper contact between the SARCs and the underlying dentin, interfering in the bond strength of the resin cement. [8,9] Thereby, the choice of acidic agents to remove the superficial loosely bound fraction of the smear layer could enhance adhesion. [8,10]

Due to the similarity of the mechanism adhesion of SARCs to glass ionomer, the polyacrylic acid has been recommended as an alternative pretreatment of root dentin. [10,11] The polyacrylic acid is a mild acid (pH=2) that contains numerous carboxyl ion groups that can form a variety of hydrogen bonds and can partially removes the smear layer, forming a thin hybrid layer to improve adhesion. [10,11] Pavan and co-authors observed that 26% polyacrylic acid improved the microtensile bond strength and could be incorporated as a cleaning step. [11] But despites of the positive impact on the bonding performance of polyacrylic acid, there are conflicting results in the literature. [8]

Another compound that has been studied is the titanium tetrafluoride (TiF_4). This fluoride compound is classified as intermediately strong acid (pH=1,5) that has a strong bond between the titanium and the oxygen atom from a phosphate group, forming a resistant vitreous layer to acids and alkaline solutions. [12-19] Since the SARCs has acidity monomers in the composition that has phosphoric acid groups, it seems that the incorporation of TiF_4 in the hybrid layer may play an important role during the adhesion procedure, but this hypothesis has never been studied.

However, some authors have been studying the application of the titanium tetrafluoride on dentin and self-etch adhesives. Dündar et al. proposed the application of an aqueous TiF_4

solution for 60s on dentin before acidic monomer application of adhesives. [17] Also, Basting et al., evaluated dentin treated with 2.5% titanium tetrafluoride and verified that this compound may modify the micromorphology of the dentin surface and form an acid resistant layer that could not be removed, even after the use of citric acid for 30 mim. [18] Lastly, Tranquilin et al. observed that a hybrid layer with the formation of more numerous and larger diameter tags, showing the favorable effects of using 2.5% TiF_4 as a pretreatment before the application of a two-step adhesive system to dentin. [14]

Therefore, a simple previous step with acidic solutions as dentin pretreatment before the luting procedure could contribute to improve mechanical properties of SARCs of SARCs, thus, the objective of this *in vitro* study was to evaluate the influence of alternative pretreatments on intra-radicular dentin bond strength of self-adhesive resin cements in fiber glass post cementation, besides of failure mode, surface morphology and adhesive interface. The null-hypotheses were that: (1) the bond strength of both SARCs would not be influenced by the pretreatment technique applied among different thirds of the intra-radicular dentin root; (2) the failure mode would not be influenced by dentin pretreatments; and (3) the hybrid layer with resin tags formation would not be influenced by the pretreatments used.

2. Materials and Methods

2.1 Specimen Preparation

Sixty extracted bovine incisors were selected, and the roots were separated from the crowns in a uniform length of 16 mm. A step-back preparation technique was used for the endodontic treatment and all the procedure were irrigated with 1% NaOCl. The roots were obturated with gutta-percha (Dentsply, Maillefer, Ballaigues, Switzerland) and AH26 sealer (Dentsply, Caulk, Milford, Germany) using the lateral condensation technique. After 48 hours, 12 mm of the endodontic material was removed and the fiber glass post space were standardized using a drill (Drill #3 - Whitepost DC, FGM, Joinville, SC, Brazil) corresponding the size of the FRC, from the same manufacturer. [20]

2.2 Luting procedure

All bovine roots were randomly allocated into six groups (n=10). Two self-adhesive resin cements were used in this study: Maxcem Elite (Kerr; Orange, CA, USA), and Calibra Universal (Dentsply Sirona; York, PA, USA); and two different pretreatments were evaluated: 26% Polyacrylic acid (Riva Conditioner, SDI, Victoria, AU) and 2.5% TiF_4 (Sigma Aldrich, Saint Louis, MO, USA). The groups that did not received a pretreatment were considered as controls. The cementation protocols of each treatment group are presented in table 1, and the products specifications used in table 2.

Table 1. Cementation protocol of each treatment group

Group	Pretreatment technic	Cementation Protocol
MAX (Fiberglass post + Maxcem Elite)	No pretreatment (Control)	1. Clean the post surface with ethanol and air-dry; 2. Remove the water excess from the root canal with an absorbent paper point; 3. Insert the self-adhesive resin cement with self-mixing tip supplied by the manufacturer into the root canal; 4. Position the fiberglass post in the center of the canal; 5. Remove the cement excess with a disposable brush; 6. Light-cure from the occlusal end for 40s.
CAL (Fiberglass post + Calibra Universal)		
MAX + PA (Fiberglass post + 26% Polyacrylic + Maxcem Elite	26% Polyacrylic Acid	1. Clean the post surface with ethanol and air-dry; 2. Remove the water excess from the root canal with an absorbent paper point; 3. Apply polyacrylic acid for 10 seconds; 4. Rinse thoroughly with water and remove excess water with an absorbent paper point; 5. Insert the self-adhesive resin cement with self-mixing tip supplied by the manufacturer into the root canal; 6. Position the fiberglass post in the center of the canal; 7. Remove the cement excess with a disposable brush; 8. Light-cure from the occlusal end for 40s.
CAL + PA (Fiberglass post + 26% Polyacrylic + Calibra Universal)		
MAX + TiF_4 (Fiberglass post + 2.5% TiF_4+ Maxcem Elite)	2.5% of titanium tetrafluoride (TiF_4) solution	1. Clean the post surface with ethanol and air-dry; 2. Remove the water excess from the root canal with an absorbent paper point; 3. Apply 2.5% TiF_4 solution for 1 minute; 4. Remove excess water with an absorbent paper point; 5. Insert the self-adhesive resin cement with self-mixing tip supplied by the manufacturer into the root canal; 6. Position the fiberglass post in the center of the canal; 7. Remove the cement excess with a disposable brush; 8. Light-cure from the occlusal end for 40s.
CAL + TiF_4 (Fiberglass post + 2.5% TiF_4+ Calibra Universal)		

Table 2: Products specifications, as reported by the manufacturer.

Material (#lot number)	**Type**	**Composition**	**Light Cure time (seconds for each surface)**	**Manufacturer**
Maxcem Elite (#7293230)	Self-adhesive resin cement	GDM, UDMA, 1,1,3,3-tetramethylbutyl hydroperoxide, TEGDMA, fluoroaluminosilicate glass, GPDM, barium glass filler, fumed silica (46 vol %)	10s	Kerr (Orange, CA, USA)
Calibra Universal (#180108)	Self-adhesive resin cement	UDMA, TMPTMA, Bis-EMA, TEGDMA, 3-(acryloyloxy)-2-hydroxypropyl methacrylate, urethane, modified Bis-GMA, PENTA, silanated barium glass, fumed silica (48 vol %)	10s	Dentsply Sirona (York, PA, USA)
Riva Conditioner (#181128)	Mild solution of polyacrylic acid	Acrylic acid polymers	Not Applied	SDI (Bayswater, VIC, AU)
2.5% TiF_4 (#MKCF9874)	Aqueous solution	2.5% of titanium tetrafluoride and distilled deionized water	Not Applied	Sigma Aldrich (Saint Louis, MO, USA)

Legend: GDM; Glycerol 1,3-dimethacrylate. UDMA; Urethane dimethacrylate. TEGDMA; Triethylene glycol methacrylate. GPDM; Glycerol phosphate dimethacryalte. TMPTMA; Trimethylolpropane trimethacrylate. Bis-EMA; Bisphenol A ethoxylate dimethacrylate. PENTA; Dipentaerythritol penttacrylate monophosphate.

The fiber glass post surface was cleaned with ethanol and air-dried before the luting procedure. For the control groups, the luting procedure was realized according to manufacturer's instructions (table 1) and light cured (Valo, Ultradent Products Inc; S. Jordan, UT, USA) from the occlusal end, with 1200mw/cm² light irradiance for 40s. The groups that received a pretreatment, a step was added before the cementation. The 26% polyacrylic acid was applied for 10 seconds, rinsed with distilled water and the excess moisture was removed with absorbent paper points according to manufactures instructions. The TiF_4 P.A. (pro-analysis) was weight in an analytical scale (OHAUS Corp, Adventure, Parsippany-Troy Hills, NJ, USA), dissolved in deionized distilled water to achieve a concentration of 2.5% (wt/v). Afterwards, the root surface was pretreated for 1 minute as used in previous studies. [13-19]

The pH of solutions were measured using a digital pHmeter (An2000; Analion, Ribeirão Preto, SP, Brazil) before the application.

2.3 Push-out bond strength test

After 7 days of the luting procedure and stored in relative humidity at 37°C, the roots were sectioned (Isomet 1000, Buehler, Lake Bluff, IL, USA) into nine 1-mm-thick slices (three slices from each root thirds: cervical, medium, and apical). The push-out test was performed in a universal testing machine (Instron, Norwood, MA, USA) at a speed of 1.0 mm/min until bond failure occurred [20]. Afterwards, the fractured samples were subjected to scanning electron microscopy (SEM) analysis to determine the failure mode.

2.4 Failure mode

The fractured samples surfaces were sputter-coated with gold, analyzed at 15 Kv by SEM (JEOL JSM-T330A, JEOL Ltd., Tokyo, Japan), and evaluated at magnifications of ×100. The fracture modes were classified into 5 types: I) failure between the dentin and the self-adhesive resin cement; II) failure between the self-adhesive resin cement and the fiberglass post; III) cohesive failure of the resin cement; IV) cohesive failure of the fiberglass post; and V) mixed failure (a combination of two or more failure types). [20]

2.5 Surface morphology and interface analysis

Another 36 bovine incisors were selected and obturated described before. The roots were subdivided into two groups each (n=3) for evaluation of surface morphology and the interface analysis. For the surface morphology, after the desobturation, the roots were sliced in the coronal-apical direction using an IsoMet Low Speed diamond saw (Buehler, Lake Bluff, IL, USA) and polished with silicon carbide papers (SiC) under water to obtain a flat surface. Afterwards, the smear layer was created with 600-grit SiC and the pretreatments were applied. Subsequently, the samples were fixed with 2.5% glutaraldehyde for 12h, gradually dehydrated using ethanol in different concentrations (25%, 50%, 75%, 95% and 100%) until a critical point (Critical Point Drying, CPD 030, Balzers, Liechtenstein) as the final dehydration step, and sputter-coated with gold. Afterwards, the surface morphology of the samples was observed at x3,000 magnification by SEM.

To analyze the adhesive interface, the root canals (n=3) were submitted to luting procedure according to table 1 and after 48 hours, sectioned in the coronal-apical direction. The specimens were wet polished with 600-, 1200-, 2000- grit sic paper and polished with diamond pastes of decreasing abrasiveness (6, 3, 1 and ¼mm). In order to observe the resin tags, specimens were pre-treated with 37% phosphoric acid solution for 10s and 5% sodium hypochlorite for 5 minutes. Lastly, the specimens allowed to dry overnight in a desiccator at

37ºC. [21] Finally, the specimens were sputter coated with gold and examined at x800 magnification by SEM.

2.6 Confocal laser scanning microscopy analysis (CLSM)

Rhodamine B dye (Sigma-Aldrich, St. Louis, MO, USA) was added to SARCs at a concentration of 0.1wt%. Eighteen roots (n=3) were restored as previously described, and samples were stored in vegetable oil at 37°C for 24h to avoid water loss and/or dye dissolution. Afterwards, roots were sectioned into 1-mm thick slices and polished. [21] Then, the samples were analyzed in CLSM (TCS SP5, Leica Microsystems, Mannheim, Baden-Württemberg, Germany). It was used argon laser at 488nm and He-Ne laser at 453nm to obtain the images in fluorescence mode, grayscale, and an image formed by overlapping the micrographs. The interface images were analyzed in 63x objective lens in oil immersion. The 20x images were analyzed by root thirds (cervical, medium, apical) and imported into the Image J software (National Institute of Health, Bethesda, MD, USA) to calculate the percentage of cement penetration into the dentin. The percentage was provided by the circumference of the root canal and the delimitation of the canal walls in with the resin cement penetrated. [22]

2.7 Statistical analysis

Data were analyzed for normal distribution and homoscedasticity (Shapiro-Wilk and Levene tests) by SPSS 21.0 (SPSS 21.0, Chicago, IL, USA). PBOS and CLSM were analyzed by three-way ANOVA and Bonferroni's *post-hoc* test, with significance level set at 5%. Failure mode, surface morphology and adhesive interface were descriptively analyzed.

3. Results

3.1 Push-out bond strength (POBS)

POBS test showed statistical significance for two independent variables: root region and dentin pre-treatment ($p < 0.001$) and their interaction ($p = 0.025$). The mean of push-out bond strength (MPa) values is shown in Table 3.

Control groups exhibited no statistical differences among the thirds, regardless of the resin cement used ($p > 0.05$). However, when a pretreatment was used (with polyacrylic acid or TiF_4), the POBS values decreased statistically from cervical to apical thirds ($p < 0.044$), with similar results between medium third and all the thirds for MAX and CAL treated with TiF_4 ($p > 0.05$).

When the pretreatments were compared between them, the results showed no statistical differences for all root thirds for both resin cements ($p > 0.05$). Considering the control group, both pretreatments presented higher POBS in the cervical third when MAX was used ($p < 0.044$), moreover, when CAL was used, only polyacrylic acid presented higher values compared to control in the cervical third ($p = 0.024$). For the medium third, there was statistical difference between TiF_4 and control group when MAX was used ($p = 0.031$). No statistical differences were observed between resin cements (MAX and CAL) evaluated for each root thirds submitted to different pretreatments ($p > 0.05$).

Table 3. Mean (SD) of push-out bond strength (MPa) of self-adhesive resin cements with different dentin pre-treatments and root regions.

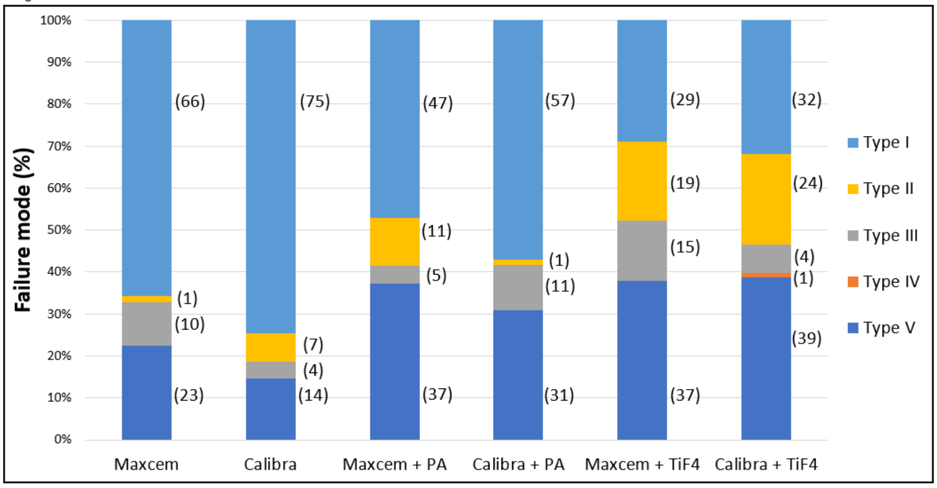
	Maxcem Elite			Calibra Universal		
	Cervical	Medium	Apical	Cervical	Medium	Apical
Control	60.31 (15.6) Ab	49.60 (18.3) Ab	48.25 (18.5) Aa	59.87 (17.7) Ab	60.51 (19.4) Aa	49.18 (29.9) Aa
26% Polyacrylic acid	77.10 (19.4) Aa	58.37 (21.0) Bab	39.80 (17.1) Ca	78.76 (16.2) Aa	50.14 (21.5) Ba	53.59 (26.1) Ba
2.5% TiF_4	79.34 (18.8) Aa	67.66 (16.8) ABa	52.49 (7.7) Ba	68.03 (14.4) Aab	62.59 (10.9) ABa	51.18 (11.6) Ba

Mean values followed by distinct letters differ statistically at 5%, according to three-way ANOVA and Bonferroni *post-hoc* test. Uppercase letters compare root thirds within dentin treatment for each resin cement (lines). Lowercase letters compare dentin treatment within each root thirds (columns). n = 10 specimens / group. Legend: TiF_4- Titanium Tetrafluoride

3.2 Failure mode

Failure modes are presented in Figure 1. For the control groups and for the polyacrylic acid pretreatment, Type-I was the most predominant failure mode followed by Type-V, regardless the resin cement. For the TiF_4, the most frequent failure mode was Type-V followed by Type-I and -II for both SARCs.

Figure 1. Failure mode

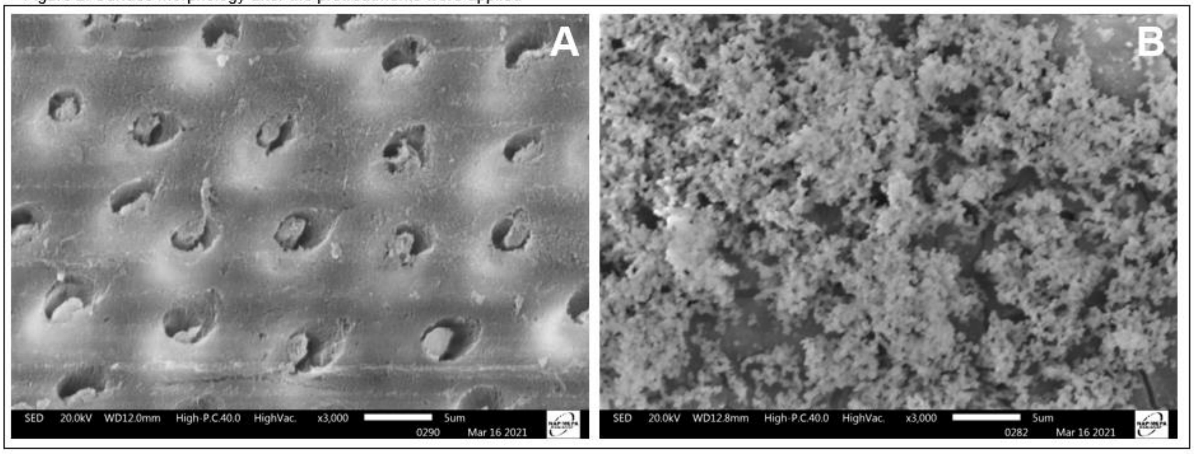


Legend: I: failure between the dentine and the resin cement; II: failure between the resin cement and the fiberglass post; III: cohesive failure of the resin cement; IV:) cohesive failure of the fiberglass post; V) mixed failure (combination of two or more failure types). PA: Polyacrylic Acid; TiF_4: Titanium Tetrafluoride (#): percentage

3.3 Surface morphology and interface analysis by SEM

The surface morphology of dentin treated with polyacrylic acid (figure 2-A) showed openned dentin tubules with mineral components in the intertubular dentin, and the surface of dentin treated with TiF_4 (figure 2-B) showed vitreous layer formed by agglomerates.

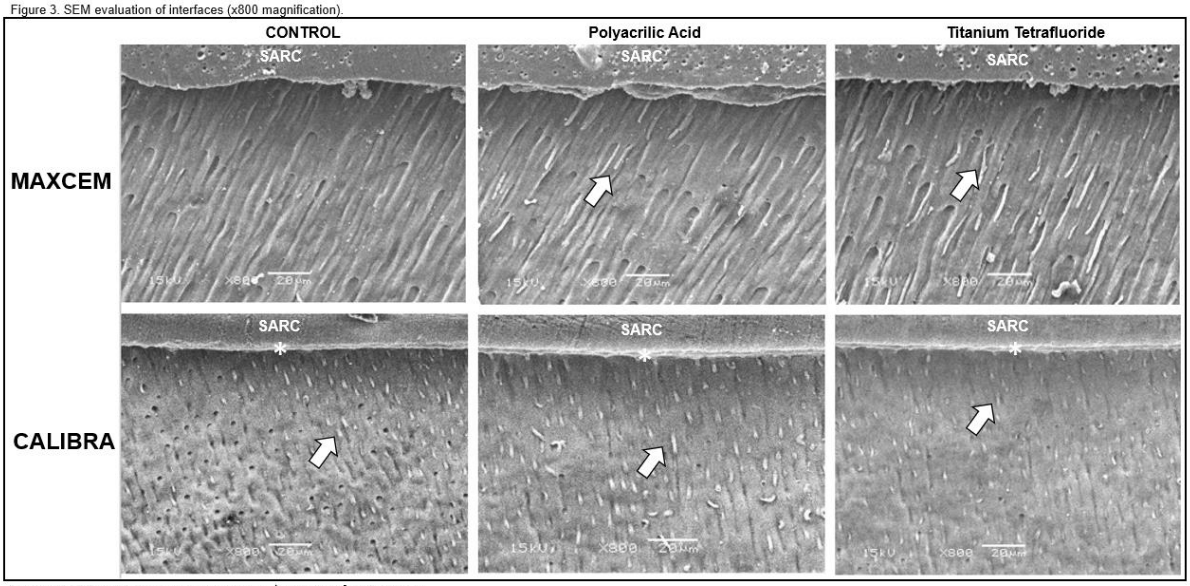
Figure 2. Surface morphology after the pretreatments were applied



Legend: A- Surface morphology after the pretreatment with Poliacrylic Acid; B- Surface morphology after the pretreatment with Titanium Tetrafluoride.

Micromorphological images (Figure 3) presented an increase of resin tags formation after the pretreatment's application, as indicated with arrows. The images revealed the formation of longer and deeper resin tags for both resin cements. For the control groups, the tags formation was visually scarce. Also, it can be observed a ticker hybrid for CAL regardless the pretreatment used, as indicated with asterisk.

Figure 3. SEM evaluation of interfaces (x800 magnification).

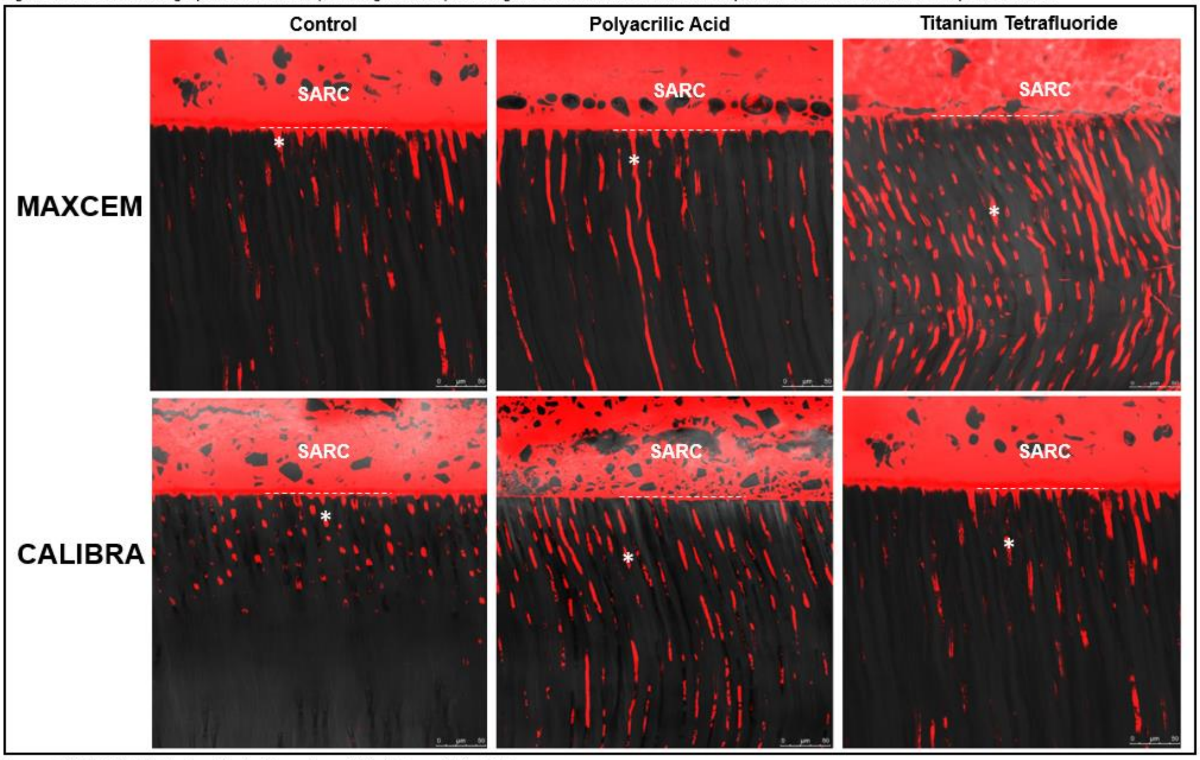


Legend: SARC: self-adhesive resin cement; ⇨ resin tag; ✱ Hybrid Layer

3.4 Confocal laser scanning microscopy analysis (CLSM)

Figure 4 shows representative images of the interface morphology analysis. It was observed hybridization zones for all the groups. It also can be observed a fewer and less deeper resin tags when the pretreatments were not applied. However, when the pretreatments were applied, a deeper penetration and longer resin tags of MAX + TiF_4 and a large quantity and shorter resin tags for CAL+ Polyacrylic Acid were observed.

Figure 4. Confocal micrographs of interfaces (x63 magnification) showing the self-adhesive resin cement penetration before and after the pretreatments.



Legend: SARC: Self Adhesive Resin Cement; ---- Hybrid Layer; * Resin Tags

The integrated density (pixels/μm^2) of the fluorescent signal obtained by CLSM (figure 5) was used to calculate the SARCs infiltration, and the values of the resin penetration in the dentinal tubules (in percentage) is presented in table 4. For the root regions, the control groups showed a decrease values from cervical to apical thirds, regardless the resin cement ($p < 0.001$). The polyacrylic acid showed no statistical differences among all thirds when CAL was used, as well as TiF_4 for MAX and CAL ($p > 0.05$). However, the apical third presented lower values when MAX was used with polyacrylic acid compared to cervical and medium thirds ($p < 0.001$).

In comparison of the pretreatments, no statistical differences were observed for cervical and medium thirds for MAX and cervical for CAL ($p > 0.05$). The control group MAX presented the highest values in the cervical third ($p < 0.001$), however, the lowest values for apical third ($p < 0.023$). Besides, control group CAL showed statistical differences between polyacrylic acid for medium third ($p < 0.030$); and the apical third presented the lowest values ($p < 0.001$).

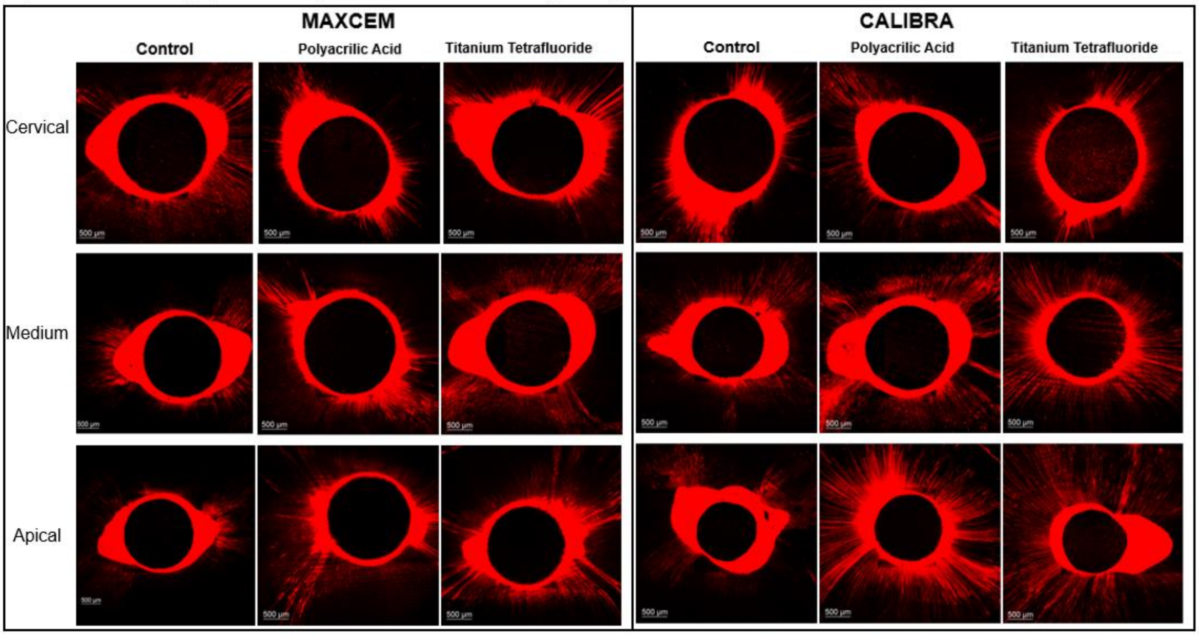
The resin cements evaluated were statistically different in all root thirds among the pretreatments ($p < 0.034$), except for control in cervical and apical thirds and TiF_4 in apical third ($p > 0.05$).

Table 4. Mean (SD) of the resin penetration in the dentinal tubules (%) of self-adhesive resin cements with different dentin pre-treatments and root regions.

	Maxcem Elite			Calibra Universal		
	Cervical	Medium	Apical	Cervical	Medium	Apical
Control	46.64 (2.9) Aa	29.99 (2.4) Ba	12.67 (9.1) Cc	45.58 (2.6) Aa	38.72 (6.1) Ab*	18.29 (10.0) Bc
26% Polyacrylic acid	36.62 (0.5) Ab	35.89 (4.5) Aa	22.13 (4.8) Bb	48.74 (1.0) Aa*	49.50 (2.9) Aa*	48.51 (7.0) Aa*
2.5% TiF_4	33.16 (0.4) Ab	33.20 (0.7) Aa	30.85 (1.2) Aa	43.35 (1.3) Aa*	41.25 (1.3) Ab*	37.36 (3.6) Ab

Mean values followed by distinct letters differ statistically at 5%, according to three-way ANOVA and Bonferroni *post-hoc* test. Uppercase letters compare root thirds within dentin treatment for each resin cement (lines). Lowercase letters compare dentin treatment within each root thirds (columns). * Compare resin cements within watch root thirds and dentin treatment. n = 3 specimens / group. Legend: TiF_4- Titanium Tetrafluoride

Figure 5. Confocal micrographs of SARCs penetration in the root thirds. Original magnification ×20.



4. Discussion

The bond strength of SARCs during the luting procedure of fiber glass posts in root canal can be impaired, since the smear layer created in root canals are denser and thicker comparing to the coronal smear layer. [6,9,15,22] Therefore, two different pretreatments to improve the demineralization and modification of the smear layer were suggested. It has been reported by several authors that TiF_4 can modify the smear layer, and polyacrylic acid can partially removes it. [10,11,16] In this study, when the pretreatments were applied, the push-out bond strength was significantly higher than the control group, considering at least one third root for each SARCs used, thus the first null hypotheses that the bond strength of both SARCs would not be influenced by the pretreatment technique applied among different thirds of the intra-radicular dentin root was rejected.

The promisor results of bond strength may be related to different mechanisms of interaction between the dentin substrate and the polyacrylic acid or TiF_4. Figure 2 shows the surface morphology after the pretreatments were applied, and the achieves corroborate with several authors [11,12,14]. The TiF_4 behaves by paring up the titanium with the oxygen atom from the phosphate group, infiltrating into the smear layer and forming agglomerates as shown in Figure 2B [12,14], and while the TiF_4 infiltrates to form a modified smear layer, the polyacrylic acid may increase the dentin permeability by removing partially the smear layer, leading to the presence of openned dentin tubules as shown in Figure 2A, but at the same time, maintaining the mineral components in the intertubular dentin, promoting a better infiltration of the SARCs. [11]

However, the increase of dentin permeability can lead to water into the root canal jeopardizing the adhesion. [8,11,23] Due to this fact, the failure type-I (failure between the dentin and the resin cement) is the most reported failure mode on the groups that received the polyacrylic acid as a pretreatment. Furthermore, the acidic monomers present in SARCs composition attracts the water due to the high hydrophilicity, and the adhesion can be impaired by poor polymerization and resin hydrolyses [3,5,6], explaining the most reported failure type I also for the control group. Yet, when the TiF_4 pretreatment was applied, the most prevalent fracture was the type-V (mixed fracture), and this type of failure mode can be associated to the modified smear layer that made the structures involved in dentin bonding acted as a single-body complex rather than separate layers. [13,8,24]. Therefore, the second null hypothesis that the failure mode would not be influenced by dentin pretreatments also needs to be reject.

Yet, to achieve good adhesion, the SARCs needs to be capable to infiltrate in the open dentin tubules, and the quantity and size of particles present in the composition it can result in an increase or decrease of viscosity. [3,25] According to table 2, the manufactures affirm that MAX has 46vol% and CAL 48.7vol% of particles, so it would be expected a deeper penetration from MAX, however, table 4 showed that CAL infiltrated higher than MAX in different thirds. These results could be attributed to the type of filler content present of each resin cement besides de vol% because they are also used to neutralize the resin acidity, and every SARC has a different acidic monomer that must be neutralized while the infiltration occur. [1,3,6]

Also, despites the limitation of this study that only the consequences of the chemical interactions of the pretreatments have been evaluated, it could be assumed that the SARCs used can have a higher potential to chemically react with the substrate or it can rely mostly on the penetration of resin monomers. In the interface analyses image (figure 3), MAX showed a limited ability to form resin tags when no pretreatments were applied and it is hard to observe a hybrid layer on this cement, however, the quantity of resin tags were improved by both pretreatments, thus, it can be assumed that this resin cement relied mostly on the micromechanical retention after the modification of the smear layer. On the other hand, it can be observed that CAL presented a thick hybrid layer regardless the pretreatment, thus, it can be assumed that this resin cement relied mostly on the chemical interactions with dentin.

Furthermore, it can be observed in representative CLSM interface images (Figure 4), a high quantity of resin tags for MAX-TiF_4 and CAL-PA. As the pH potential has been associated with the mechanical behavior of SARCs [1,10, 26], it can be assumed that due to the interaction of the highly acidic pH (about 1.5) of the TiF_4 and the acid monomers present in MAX, it occurred a deeper demineralization and infiltration of the resin cement. Regarding the polyacrylic acid, it is classified as a mild solution (pH about 2), and according to Giannini and co-authors [27], mild acids has the advantage of leaving substantial amount of hydroxyapatite-crystals around collagen fibrils, which may establish chemical bond with specific carboxylic or phosphate groups of functional monomers. Therefore, it can be assumed that the functional monomer present in CAL composition had a better chemical interaction with a mild pH when compared to the intermediate-strong pH of the TiF_4.

The SARCs evaluated in this study has different acidic monomers group, such as GPDM (glycero-phosphate dimethacrylate) in MAX and PENTA (dipentaerythritol penta-acrylate phosphate) in CAL. The hydrophilic monomer GPDM has a good denting wetting permeability, stability and can be efficiently polymerized with other co-monomers creating a stronger polymer network due to the two methacrylate groups [28]. Otherwise, PENTA monomer contains a more viscous molecule and a shorter main chain with five vinyl groups [29]. Thus, the combination of the pH, the acidic monomer, and particles such as: quartz, colloidal silica, and glass fillers, are important to discuss the mechanism of adhesion, however, the specific composition and concentrations of each component are not available in the manufacture's instruction. [13,28,29]

But despite the scarce information of the specific composition, the ability of the pretreatments to modify or partially removes the smear layer, improved the homogeneity infiltration of the SARCs in all thirds according to the quantitative analysis of percentage penetration of resin cement (pixels/µm2) present in table 4, therefore, the third null hypothesis that the hybrid layer with resin tags formation would not be influenced by the alternative pretreatments was rejected.

The Figure 5 shows a representative image of SARCs penetration. The control groups showed a decrease in resin tags infiltration along the thirds as expected, since the conformation of root canals turns the adhesion procedure more difficult, but when the pretreatments were applied, it resulted in the modification of the smear layer and allowed a deeper resin infiltration. The cervical third has more tubule density and a larger diameter as reported in the literature [9, 22], explaining the large quantity of resin tags formation in all the groups. But the apical third has more sclerotic dentin and less tubule density than the coronal third [9, 22], yet it can be observed an increase of resin tags when PA or TiF_4 were applied compared to control, regardless the resin cement used, however, deeper resin tags may occupy the tubule without adhering to the walls, and polymerization shrinkage may produce hollow resin tags. [22,24,30] Therefore, the presence of deep resin tags does not add much to bond strength, because the main factor that contributes with the resin cement-dentin bond is the entanglement between resin cement and intertubular collagen fibrils bond. [24,30].

Despite the advances in developing resin cements, the mechanism of adhesion in root canals is still a challenge, and the smear layer can be considered the weakest link in the luting procedure; besides, the presence of water in the dentin tubular and the excessive hydrophilicity of SARCs could compromise the mechanical stability [1,3,4,5], thus, longitudinal studies are recommended. But as an advantage, dentin pretreatments are a simple and fast clinical procedure that has shown positive results by modifying the smear layer improving mechanical properties of this category of resin cements.

5. CONCLUSION

The application of acid solutions as dentin pretreatment before self-adhesive resin cement insertion, with polyacrylic acid or 2.5% TiF_4 solution, were able to modify the smear layer improving the push-out bond strength values. And depending on the SARC composition, the pretreatments were capable to form longer, deeper, and homogeneous resin tags infiltration among the root thirds.

COMPLIANCE WITH ETHICAL STANDARDS

Conflict of Interest:

Marina Rodrigues Santi declares that she has no conflict of interest.
Rodrigo Barros Esteves Lins declares that he has no conflict of interest.
Beatriz Ometto Sahadi declares that she has no conflict of interest.
Jorge R. Soto-Monteiro declares that he has no conflict of interest.
Luís Roberto Marcondes Martins declares that he has no conflict of interest.

Ethical approval: This article does not contain any studies with human participants or animals performed by any of the authors.
Informed consent: For this type of study, formal consent is not required.

FIGURE LEGENDS

Fig.1 Failure mode.
Legend: type-I: failure between the dentin and the self-adhesive resin cement; type-II: failure between the self-adhesive resin cement and the fiberglass post; type-III: cohesive failure of the resin cement; type-IV: cohesive failure of the fiberglass post; type-V: mixed failure (combination of two or more failure types). PA: Polyacrylic Acid; TiF_4: Titanium Tetrafluoride.

Fig.2 Surface morphology after application of the pretreatments.
Legend: A- Surface morphology after the pretreatment with Polyacrylic Acid; B- Surface morphology after the pretreatment with Titanium Tetrafluoride.

Fig.3 SEM evaluation of interfaces (x800 magnification).
Legend: SARC: self-adhesive resin cement; T: tag; HL: Hybrid Layer

Fig.4 Confocal micrographs of interfaces (x63 magnification) showing the self-adhesive resin cement penetration before and after the pretreatments.
Legend: SARC: Self-Adhesive Resin Cement; ⇨ Resin Tags; * Hybrid Layer

Fig.5 Confocal micrographs of SARCs penetration in the root thirds. Original magnification ×20.

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3 CONCLUSÃO

Os cimentos resinosos auto-adesivos foram desenvolvidos para diminuir o número de passos clínicos, porém a smear layer criada durante o preparo de canais radiculares para a cimentação de pinos de fibra de vidro, pode ser considerada um fator prejudicial a adesão devido a sua espessura ser mais grossa comparada a smear layer coronária. Portanto, apesar das limitações deste estudo, deve-se considerar a aplicação de uma solução ácida de 2,5 TiF_4 ou 26% de ácido poliacrílico como pré-tratamento prévio a cimentação, pois estas soluções foram capazes de modificar a smear layer melhorando a resistência de união no terço cervical e, dependendo da composição do cimento utilizado, foram capazes de melhorar a penetração dos cimentos, deixando-os mais homogéneos e formando uma maior quantidade de tags resinosos.

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ANEXOS

Anexo 1 - Verificação de Originalidade e Prevenção de Plágio

Evaluation of alternative pretreatments for dentin on bond strength of self-adhesive resin cements

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