



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



Giovanna Righetti Bravo

**Efeitos do hipoclorito de cálcio na interface de adesão entre a dentina radicular
e o sistema de cimentação de pinos de fibra**

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Tese apresentada à Universidade Estadual Paulista (Unesp), Faculdade de Odontologia, Araraquara para obtenção do título de Doutora em Nome do Programa de Odontologia, na Área de Endodontia

Orientador: Milton Carlos Kuga

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Giovanna Righetti Bravo

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Comissão julgadora

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Araraquara, 05 de setembro de 2022.

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RESUMO

Este estudo avaliou os efeitos do hipoclorito de sódio (SH) e do hipoclorito de cálcio (CH) quando submetidos à irrigação ultrassônica passiva (PUI) ou à irrigação convencional (IC) sobre a persistência de resíduos, a resistência de união e o padrão de fratura de um sistema de cimentação resinoso à dentina intrarradicular do espaço preparado para pino de fibra. Também foi avaliado o efeito dessas substâncias na remoção de 0,01% do azul de metileno, após PDT, com um sistema de cimentação resinoso e um cimento de ionômero de vidro. Duzentos e dez incisivos bovinos foram tratados endodonticamente e preparados para pino de fibra. Dentre esses, 120 dentes foram aleatoriamente divididos em 6 grupos de acordo com o método de solução e irrigação: DW-CI, SH-ED-CI, SH-CI, SH-PUI, CH-CI e CH-PUI. Após os protocolos de irrigação a persistência de resíduos sobre a dentina intrarradicular do espaço preparado para pino foi avaliada por imagens SEM. Foi realizada então a cimentação dos pinos de fibra utilizando um cimento resinoso convencional dual (RelyX Arc) e um sistema adesivo de 2 passos. O teste de push-out foi realizado para a avaliação da resistência de união e o padrão de fratura também foi avaliado. Outros noventa dentes bovinos foram submetidos ao PDT utilizando 0,01% azul de metileno e irradiados com laser de diodo (660 nm). Três protocolos de irrigação foram testados para remover o azul de metileno por incidência de resíduos sobre dentina (n=10): água destilada (DW), hipoclorito de sódio de 2,5% (SH) e hipoclorito de cálcio de 6% (CH). Os outros setenta dentes foram usados para avaliar a resistência de união à dentina intrarradicular de acordo com o protocolo de irrigação e o sistema de cimentação utilizado (Cimento RelyX U200 (RU) e cimento ionômero de vidro GC Gold Label 1 (GC) em 6 grupos (n=10): DW-RU, SH-RU, CH-RU, DW-GC, SH-GC e CH-GC.

Resultados: Os dados não paramétricos e paramétricos foram analisados a um nível de significância de 5%. SH-ED-CI, SH-PUI e CH-PUI apresentaram menor incidência de resíduos para o terço cervical e SH-ED-CI para o terço médio ($p < 0,05$). Os valores de resistência de união foram mais elevados após a irrigação com DW-CI para todos os terços ($p < 0,05$). Independentemente do terço radicular a DW apresentou menor incidência de resíduos do que a SH ($p < 0,05$), mas semelhante ao CH ($p > 0,05$). Por outro lado, a SH apresentou resultados semelhantes ao CH ($p > 0,05$). Em relação à resistência de união à dentina, CH-RU e CH-GC apresentaram valores mais elevados em relação aos outros grupos ($p < 0,05$), mas semelhantes entre si ($p > 0,05$). Não foram observadas diferenças entre os demais grupos ($p > 0,05$). O padrão de fratura coesiva foi predominante.

Conclusões: tanto o hipoclorito de sódio quanto o hipoclorito de cálcio a 2,5% impactam negativamente a interface de adesão e apresentam maior incidência de resíduos sobre a dentina radicular no espaço preparado para pino. Contudo, o hipoclorito de cálcio a 6% na remoção de 0,01% azul de metileno após PDT favorece a resistência de união de RU e GC à dentina intrarradicular do espaço preparado para pino de fibra.

Palavras chave: Hipoclorito de cálcio. Hipoclorito de sódio. Adesividade. Dentina.

BRAVO GR. Effects of calcium hypochlorite on the adhesion interface between root dentin and fiber post cementing system [tese de doutorado]. Araraquara: Faculdade de Odontologia da UNESP; 2022.

ABSTRACT

This study evaluated the effects of sodium hypochlorite (SH) and calcium hypochlorite (CH) when submitted to passive ultrasonic irrigation (PUI) or conventional irrigation (CI) on residue persistence, union resistance and fracture pattern of a resin cementation system to intraradicular dentin from the fiber post space. The effect of these substances on the removal of 0.01% of methylene blue after PDT was also evaluated with a resin cementation system and a glass ionomer cement. Two hundred and ten bovine incisors were endodontically treated and prepared for fiber post. Among these, 120 teeth were randomly divided into 6 groups according to the solution and irrigation method: DW-CI, SH-ED-CI, SH-CI, SH-PUI, CH-CI and CH-PUI. After the irrigation protocols, the persistence of residues on the intraradicular dentin of the post space was evaluated by SEM images. The fiber posts were cemented using a conventional dual resin cement (RelyX Arc) and a 2-step system. The push-out test was performed to evaluate the union resistance and the fracture pattern was also evaluated. Ninety other bovine teeth were submitted to PDT using 0.01% methylene blue and irradiated with diode laser (660 nm). Three irrigation protocols were tested to remove methylene blue by incidence of dentin residues (n=10): distilled water (DW), sodium hypochlorite of 2.5% (SH) and calcium hypochlorite of 6% (CH). The other seventy teeth were used to evaluate the union resistance to intraradicular dentin according to the irrigation protocol and the cementation system used (RelyX U200 Cement (RU) and GC Gold label 1 (GC) glass ionomer cement in 6 groups (n=10): DW-RU, SH-RU, CH-RU, DW-GC, SH-GC and CH-GC.

Results: Nonparametric and parametric data were analyzed at a significance level of 5%. SH-ED-CI, SH-PUI and CH-PUI showed a lower incidence of residues for the cervical third and SH-ED-CI for the middle third ($p < 0.05$). The values of union resistance were higher after irrigation with DW-CI for all thirds ($p < 0.05$). Regardless of the root third, DW had a lower incidence of residues than SH ($p < 0.05$), but similar to CH ($p > 0.05$). On the other hand, HS presented results similar to CH ($p > 0.05$). Regarding dentin union resistance, CH-RU and CH-GC presented higher values in relation to the other groups ($p < 0.05$), but similar to each other ($p > 0.05$). No differences were observed between the other groups ($p > 0.05$). The cohesive fracture pattern was predominant.

Conclusions: both sodium hypochlorite and 2.5% calcium hypochlorite negatively impact the beforehand interface and have a higher incidence of residues on root dentin in the pin-prepared space. However, the 6% calcium hypochlorite in the removal of 0.01% methylene blue after PDT favors the favor resistance of RU and GC union to the intraradicular dentin of the space prepares for fiber post.

Keywords: Calcium hypochlorite. Sodium hypochlorite. Adhesiveness. Dentin.

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1 INTRODUÇÃO

Os pinos de fibra de vidro estão indicados para situações clínicas com significativa perda de estrutura da coroa dental e que necessitam de retentores intracanaís para possibilitar a reabilitação do elemento^{1,2}. Sua escolha deve-se principalmente às suas características de similaridade ao módulo de elasticidade da dentina, relativa simplicidade técnica ao permitir a realização imediata pelo profissional em consultório e a adesão a materiais resinosos. Entretanto, também é necessário que sua adesão ao substrato dentinário seja efetiva para permitir a longevidade do tratamento¹⁻⁴.

Fatores como a escolha da solução de limpeza, o tipo de cimento endodôntico utilizado na obturação o tratamento adequado da superfície dentinária com controle da umidade, a escolha do sistema adesivo, bem como a correta inserção e polimerização do cimento, demonstram a sensibilidade da técnica e podem resultar em insucesso.⁵⁻⁷ Além disso, a contaminação deste local por microrganismos oriundos da própria cavidade bucal em casos de microinfiltração por falhas adesivas pode não somente comprometer o tratamento restaurador como também comprometer o tratamento dos canais radiculares resultando em insucesso endodôntico⁸⁻¹⁰

Adicionalmente, há a formação de uma camada de debris constituída por partículas dentinárias, cimento endodôntico e guta percha plastificada, que é produzida pelo calor das brocas durante o preparo do conduto para o pino e oblitera os túbulos dentinários¹¹⁻¹³. Em decorrência disso, o padrão de falha nos protocolos de reabilitação utilizando pinos de fibra é predominante na interface entre cimento e dentina, ocasionando a soltura do pino^{5,14}.

Por isso, diversos protocolos de irrigação são propostos com o objetivo de remover esses debris e expor as fibras colágenas para permitir a formação da camada híbrida, ao mesmo tempo em que descontamina o espaço preparado para pino, favorecendo uma adesão efetiva aos dois substratos¹⁵⁻²⁰.

Rotineiramente é recomendada a irrigação com a associação da solução de hipoclorito de sódio ao ácido etilenodiamínicotetracético (EDTA) para essa finalidade^{21,22}. Entretanto, essa associação parece exercer efeitos negativos sobre a adesão e comprometer a longevidade do tratamento. A solução de hipoclorito de sódio a 2,5% é degradada em hidróxido de sódio e ácido hipocloroso (HOCl)²³. Por

sua vez, durante a catálise química do HOCl ocorre a liberação de oxigênio singlet, que pode interferir negativamente sobre o mecanismo de polimerização por competirem com os radicais livres na reação de polimerização dos metacrilatos e ocuparem fisicamente os espaços durante a formação da camada híbrida, o que aumenta os riscos de microinfiltração e pode interferir negativamente também sobre a interface de adesão do sistema de cimentação resinoso convencional com o adesivos utilizados na estratégia etch-and-rinse^{15,24-26}.

Para a cimentação de pinos de fibra podem ser utilizados protocolos distintos, utilizando os cimentos resinosos convencionais, com ou sem cura dupla, ou os autoadesivos, que podem ser utilizados através do sistema adesivo condiciona e lava ou autocondicionante^{15,27}. Ainda não há um consenso sobre qual o melhor protocolo, mas estudos prévios mostram resultados de retenção superiores para protocolos que utilizam cimentos de resina de polimerização dual associados ao sistema adesivo condiciona e lava² e para protocolos que utilizam cimentos resinosos autocondicionantes associados ao sistema adesivo condiciona e lava, entretanto, o segundo parece ser mais vulnerável devido à dificuldade em fazer com que a luz penetre por todo o espaço para pino, o que interfere negativamente na sua polimerização⁷.

Com o objetivo de minimizar os efeitos indesejáveis do NaOCl, o hipoclorito de cálcio ($\text{Ca}(\text{OCl})_2$), que é uma solução amplamente utilizada na esterilização e purificação industrial de água, apresenta propriedades interessantes para ser utilizado na odontologia. Dentre elas estão satisfatória atividade antimicrobiana, maior citocompatibilidade e estabilidade, além de eficácia na dissolução de tecidos orgânicos, porém com uma taxa mais lenta quando comparada ao NaOCl ³⁰⁻³⁷. Apesar de haver maior liberação de cloro durante sua catálise química, apresenta maior tensão superficial do que o NaOCl ^{32,33,38}. Consequentemente, ainda são controversos os seus efeitos quando utilizado como na irrigação do espaço intrarradicular e sua influência na adesão dos novos sistemas resinosos de cimentação para pino de fibra de vidro.

Adicionalmente, a irrigação ultrassônica passiva (PUI) tem efeito sinérgico sobre as propriedades de limpeza da superfície dentinária das soluções de irrigação endodôntica pois promove seu aquecimento e permite o alcance a regiões intocadas pelos instrumentos rotatórios. Além disso, parece potencializar a ação de dissolução

do NaOCl, removendo mais detritos orgânicos e inorgânicos das paredes dentinárias^{31,39-41}.

A terapia fotodinâmica (PDT) também é uma terapia coadjuvante com potencial antimicrobiano, reduzindo a carga de microrganismos dentro do canal radicular⁴². A atividade antimicrobiana ocorre devido à interação entre uma fonte de luz e um foto sensibilizador, como 0,01% azul metileno, que promove a formação e liberação de radicais oxidativos, como o oxigênio singlet, causando dano/morte celular^{43,44}.

Entretanto, o uso de corantes como o azul metileno pode mudar a cor do dente, comprometendo procedimentos estéticos de reabilitação⁴⁵⁻⁴⁸. Para minimizar esses efeitos indesejáveis, vários protocolos têm sido recomendados, principalmente utilizando soluções oxidativas para clarear o tecido, como hipoclorito de sódio (NaOCl) em diferentes concentrações^{48,49}. No entanto, como durante a dissociação química de NaOCl há a formação de hidróxido de sódio e ácido hipocloroso, que afetam negativamente a interface de ligação com sistemas convencionais de cimentação com adesivos de gravura e lavagem^{15, 49}. Considerando essas deficiências, foi proposital o uso de 6% de solução de hipoclorito de cálcio ($\text{Ca}(\text{OCl})_2$), que também tem a capacidade de esclarecer tecidos, foi proposital⁵¹. Embora o $\text{Ca}(\text{OCl})_2$ tenha cloro em sua composição, observa-se maior estabilidade química em relação ao hipoclorito de sódio, permitindo uma dissociação iônica mais gradual que tende a minimizar o impacto na interface adesiva com cimento de resina convencional^{31,32,50}.

Para otimizar o tempo clínico e minimizar erros durante a pós cimentação de fibras, o cimento de resina autoadesiva e os ionômeros de vidro têm sido usados sobre cimento convencional de resina^{15,51}. A adesão desses cimentos ocorre por meio de ligações entre os componentes químicos do sistema de cimentação com o cálcio da hidroxiapatita do substrato dentinário^{51,52}. Por outro lado, a solução de hipoclorito de cálcio dissocia em hidróxido de cálcio, que precipita na superfície da dentina. No entanto, os efeitos da interação entre esses resíduos e os sistemas de cimentação ainda são desconhecidos³².

Levando em consideração essas informações, foi observada a necessidade de encontrar um protocolo que remova adequadamente os debris da superfície dentinária para deixar os túbulos dentinários livres para uma adesão efetiva, mas sem interferir na resistência de união do sistema de cimentação de pinos de fibra na dentina radicular⁵³.

Uma vez que os estudos sobre a remoção de debris pela associação do NaOCl à PUI são inconclusivos, poucos estudos sobre a remoção de debris pelo Ca(OCl₂) estão disponíveis e são também contraditórios, achamos oportuno avaliar a interação dos protocolos ora descritos através de métodos que avaliem seus efeitos e eficiência^{15,54-56}.

2 PROPOSIÇÃO

Este estudo teve como objetivo avaliar os efeitos do hipoclorito de cálcio ou sódio (2,5%) ativados ou não com PUI na interface adesiva. Para isso, foram avaliadas a presença de resíduos e a resistência de união à dentina após a irrigação do espaço preparado para pino nos terços cervical, médio e apical. Os procedimentos de adesão foram realizados utilizando-se um cimento resinoso convencional dual (RelyX ARC) com um sistema adesivo de 2 passos (Single Bond 2). As hipóteses nulas testadas foram: H01 - não há correlação favorável entre os protocolos de irrigação e a incidência de resíduos na superfície da dentina nos diferentes terços; H02 - não há correlação favorável entre os protocolos de irrigação e a resistência de união do cimento resinoso convencional dual e do sistema adesivo de 2 passos na superfície dentinária nos diferentes terços.

Este estudo também avaliou os efeitos da remoção de 0,01% de azul de metileno após PDT na dentina intrarradicular do espaço preparado para pino usando irrigação com água destilada, solução de hipoclorito de sódio de 2,5% ou hipoclorito de cálcio de 6%. A resistência de união à dentina e o modo de falha após a cimentação de pinos de fibra de vidro com cimento de resina autoadesiva (Relyx U200) ou cimento ionômero de vidro (GC Gold Label 1 Luting & Lining Cement) em terços cervicais, médios e apical do espaço pós-escola também foi avaliado. As hipóteses nulas testadas foram: H01 - Não há diferença entre os protocolos de irrigação na incidência de resíduos na superfície da dentina; H02 - Não há diferença na força de ligação e no modo de falha entre os sistemas de cimentação.

3 PUBLICAÇÕES

É resultado desta pesquisa os artigos apresentados a seguir.

3.1 Publicação 1*

Bonding and cleaning effects of irrigation protocols using calcium hypochlorite on the post-space radicular dentin

ABSTRACT

Introduction: This study evaluated the effect of 2.5% sodium hypochlorite (SH) or calcium hypochlorite (CH) submitted to passive ultrasonic irrigation (PUI) or conventional irrigation (CI) on the incidence of residues and the bond strength of the cementation system to post-space dentin. **Methods:** 120 bovine incisors were endodontically treated and post-space preparation was performed. Distilled water (DW) and 2.5% SH followed by 17% EDTA (SH-ED) were used as control groups. The specimens were randomly assigned to 6 groups, according to the solution and irrigation method: DW-CI, SH-ED-CI, SH-CI, SH-PUI, CH-CI, and CH-PUI. Incidence of residues over the post-space dentin was evaluated by SEM images. After the irrigations protocols, post cementation was immediately performed using a conventional dual resin cement and a 2-step etch-and-rinse adhesive system. Push-out and failure mode were performed for bonding evaluation. Non-parametric and parametric data were analyzed at a significance level of 5%. **Results:** SH-ED-CI, SH-PUI and CH-PUI showed lower incidence of residues for cervical third and SH-ED-CI for middle third ($p < 0.05$). In the apical third, the protocols were similar to each other ($p > 0.05$). Bond strength values were higher after irrigation with DW-CI for all thirds ($p < 0.05$). **Conclusions:** 2.5% sodium or calcium hypochlorite negatively impacted the adhesion interface and exhibit greater incidence of residues over the post-space radicular dentin.

Keywords: calcium hypochlorite; adhesion; bond strength; irrigation; sodium hypochlorite

* Artigo elaborado seguindo as normas do periódico *Operative Dentistry*, para o qual o trabalho foi submetido (ANEXO A).

INTRODUCTION

The root canal may be contaminated by microorganisms from saliva during the post space preparation¹, which highlights the need for disinfection strategies. Thus, to reduce or eliminate contamination inside the root canal, irrigation using solutions with antimicrobial activity and the ability to remove the residues over the dentin without negatively affect the bonding interface is crucial and indispensable^{2,3}.

Commonly, 2.5% sodium hypochlorite (NaOCl) solution is widely used to irrigate root canals. This solution dissociates into sodium hydroxide and hypochlorous acid (HOCl)^{4,5}. However, the chemical catalysis of HOCl generates molecules of singlet oxygen which may negatively impact the adhesive infiltration into the dentin and the adhesive polymerization since the oxygen molecules compete with oxidative radicals that are responsible for the polymerization process of methacrylate chains in resin-based materials^{4,6,7-9}.

Several substances were proposed to replace NaOCl as root canal irrigants which include chlorhexidine or chemical associations with acids and/or surfactants^{1,5}. However, these substances have not demonstrated successful antimicrobial activity and/or suitable cleaning potential of the dentin surface, besides promoting deleterious effects on bonding interface^{10,11}.

Calcium hypochlorite ($\text{Ca}(\text{OCl})_2$) is a chlorine-based alternative that is also used as antimicrobial solution on water purification and disinfection in basic sanitation practices¹². $\text{Ca}(\text{OCl})_2$ has shown similar antimicrobial activity, enhanced chemical stability and better tissue tolerance when compared to NaOCl^{13,14,7}. In the chemical catalysis of $\text{Ca}(\text{OCl})_2$, gradual release of oxidative radicals is verified, which can be beneficial to reduce possible negative effects on the polymerization reaction of dual resin cements⁷⁻⁹.

Although the advantages previously mentioned, $\text{Ca}(\text{OCl})_2$ does not appear to provide suitable removal of the smear layer, which is the amorphous superficial layer formed by instrumentation of the root canals⁸. Particularly in post space preparation, the cleaning of dentin surface is even more challenging since the smear layer formed is composed by obturation residues and/or dentin debris¹⁵. To overcome the limitations of $\text{Ca}(\text{OCl})_2$ and to enhance its clinical performance, the ultrasound activation of the solutions by passive ultrasonic irrigation (PUI) has been recommended¹⁶.

PUI exerts a synergic cleaning effect on the dentin surface since PUI promotes greater irrigation flow, low heating, and enhanced diffusion of the irrigation solution into

areas hard to reach by rotatory instruments. Furthermore, PUI appears to enhance the NaOCl dissolution, removing more debris due to the acoustic microstreaming and hydrodynamic cavitation. When compared to conventional irrigation, PUI is significantly better to remove organic residues, optimizing the disinfection of the root canals and the post space^{7,17-19}.

However, the effects of irrigation protocols using $\text{Ca}(\text{OCl})_2$ after post space preparation are unclear and deserve further investigations. In addition, the PUI effectiveness of enhance the cleaning capacity in comparison to NaOCl alone or associated with 17% EDTA is still uncertain. It is worth to mention that the effects of $\text{Ca}(\text{OCl})_2$ on the bonding interface between the root dentin and the conventional dual resin cement with etch-and-rinse adhesive system remain unknown²⁰.

This study aimed to evaluate the effect of calcium or sodium hypochlorite (2.5%) activated or not with PUI on the adhesive interface. For this, presence of residues and bond strength to dentin after post space preparation at cervical, middle and apical thirds were evaluated. The bonding procedures were performed using a conventional dual resin cement (RelyX ARC) with 2-step etch-and-rinse adhesive system (Single Bond 2). The null hypotheses tested were that: H_01 - there is no positive correlation between the irrigation protocols and the incidence of residues on the dentin surface at the different thirds; (H_02), there is no positive correlation between the irrigation protocols and the bond strength of the conventional dual resin cement and etch-and-rinse adhesive system on the dentin surface at different thirds.

MATERIALS AND METHODS

Ethical aspects

This study was properly approved by the Ethical Committee in Animal Use under the register number 19/2017.

Sample preparation

One hundred and twenty bovine conoid incisors without root anatomic defects were selected. After selection and cleaning, teeth were stored in 0.1% thymol solution at 4°C ($\pm 1^\circ\text{C}$) for 30 days until the beginning of the experiments.

The teeth were transversely sectioned using diamond-coated discs (#7020, KG Sorensen, Cotia, SP, Brazil). The root length was standardized in 17 mm from the root apex⁵. After glide path and apical patency using #15 K-file (Maillefer Dentsply,

Ballaigues, Switzerland), the apexes were sealed with glass ionomer cement (Maxxion R, FGM Produtos Odontológicos Ltda, Joinville, SC, Brazil) and the root canals were instrumented up to the F5 instrument (ProTaper, Dentsply Maillefer, Ballaigues, Switzerland), according to the manufacturer instructions. The working length was established in 16 mm.

Endodontic irrigation was performed at each instrument change using 5mL of 2.5% NaOCl (Asfer Indústria Química, São Caetano do Sul, SP, Brazil). After NaOCl aspiration, final irrigation was performed with 17% EDTA and kept inside the root canal for 3 minutes followed by rinsing with 5mL of 2.5% NaOCl. The canals were then aspirated using endodontic tips (Cappillary tips; Ultradent South Jordan, UT, USA) and dried with F5 paper points (ProTaper; Dentsply Maillefer, Ballaigues Switzerland). Root canal obturation were performed using F5 gutta percha point and epoxy resin-based sealer (AH Plus, Dentsply De Trey, Konstanz, GER) by the single-cone technique. Excess material was removed and the cervical opening was provisionally restored (Coltosol, Coltene, Rio de Janeiro, RJ, Brazil). The roots were stored in distilled water at 37°C ($\pm 1^\circ\text{C}$) for 7 days²¹.

Post-space preparation was initially performed with #2 and #3 Largo drills (Dentsply Maillefer, Ballaigues, Switzerland) to a depth of 11 mm. In sequence, a DC2 drill (White Post; FGM, Joinville, SC, BR) was used for the finishing. The drills were adapted in an endodontic handpiece (X-Smart Plus; Dentsply Sirona) at 800 rpm and a torque of 0.5 N.cm. None irrigation solution was used during post space preparation.

Irrigation protocols

Twenty obturated specimens were used for each group (n = 10 for incidence of residues and n = 10 for push out bond strength evaluation). The irrigation protocols of the post space were:

DW-CI (distilled water and conventional irrigation): irrigation with 5mL of distilled water using luer lock syringe and irrigation tips (Navitip 30G; Ultradent, South Jordan, UT, USA). The syringe was filled with 5 mL of the irrigant and the tip was placed at 10 mm inside the root canal in continuous movements from apical to cervical third. These procedures were similar employed in all protocols that used conventional irrigation;

SH-ED-CI (sodium hypochlorite with EDTA and conventional irrigation): irrigation with 5mL of 2.5% sodium hypochlorite (Asfer Indústria Química, São Caetano do Sul, SP, Brazil) followed by aspiration (Cappillary tips; Ultradent, South Jordan, UT, USA). After that, irrigation with 5mL of 17% EDTA (Biodinâmica, Ibiporã, PR, Brazil)

was performed and the solution was kept into the post space for 3 minutes. In sequence, a final rinse was performed using 2.5% sodium hypochlorite.

SH-CI (sodium hypochlorite and conventional irrigation): similar to DW-CI protocol, but using 2.5% sodium hypochlorite (Asfer Indústria Química, São Caetano do Sul, SP, Brazil);

SH-PUI (sodium hypochlorite and passive ultrasonic irrigation): initially the post space was filled with 2.5% NaOCl, and an activation tip (E1; Helse, Santa Rosa de Viterbo, SP, BR) coupled in an ultrasonic device (Varios II; NSK Company, Tochigi, JPN) was placed at 10 mm inside the root canal. The solution was activated for 3 cycles of 20 seconds each, using movements from apical to cervical third without touching the tip on the dentin surface. At each ultrasound activation time, the 2.5% NaOCl was replaced in post space¹⁹.

CH-CI (calcium hypochlorite and conventional irrigation): similar to SH-CI protocol, but using 2.5% calcium hypochlorite (IQ-Car, Araraquara, SP, Brazil). This solution was obtained by diluting 3.847 g of $\text{Ca}(\text{OCl})_2$ into 100 mL of distilled water at 30° C. Constant stirring was performed until the complete dissolution of the solute. Afterward, the solution was filtered twice to remove debris and immediately used¹³.

CH-PUI (calcium hypochlorite and passive ultrasonic irrigation): similar to SH-PUI, but using 2.5% calcium hypochlorite.

After performing all the irrigation protocols, the post space was aspirated and dried with absorbent paper points.

Incidence of residues

Sixty specimens were used and randomly assigned to the 6 study groups (n = 10). After irrigation of the post-space, longitudinal groove in the buccal and lingual surfaces of the roots was made with a double face diamond-coated disc (KG Sorensen, São Paulo, SP, Brazil). The roots were then cleaved in buccolingual direction acusing a chisel (Golgran, São Paulo, SP, Brazil) to obtain mesial and distal root sections and the distal section was processed for microscopy as described by Magro et al²².

Four areas of each third of the post space were analyzed and a representative image of the observed surface was photographed at 500x of magnification using a scanning electron microscope (LEO 435VP, Carl Zeiss Microscopy Ltd, Cambridge, UK) in 10 kV. All images were photographed by a single and independent operator blinded to the experiment.

To compare the incidence of residues, the dentin surface was graded by scores according to the amount of opening dentin tubules and surface debris as previously described by Serafino et al¹⁵. The scores were graded between 0 and 2: score 0 – all dentin tubules were open and no debris or sealer remnants covering the opening of the tubules were verified; score 1 – most of the dentin tubules were open and some debris or sealer remnants covering the opening of the tubules were verified; score 2 – most of the dentin tubules were covered and a great amount of debris or sealer remnant were verified.

Bond strength

Sixty specimens were randomly allocated to the study groups (n = 10). Previously to the evaluation, the adaptation of the fiber post (DC#2; White Post; FGM Produtos Odontológicos, Joinville, SC, Brazil) inside the root canal and its penetration through the entire length of the post space was confirmed. Then, the fiber post surface was etched with 37% phosphoric acid (Dentsply, Pirassununga, SP, Brazil) for 1 minute, profusely rinsed with distilled water and dried with air-jets. In sequence, three layers of silane (Prosil, FGM, Produtos Odontológicos, Joinville, SC, Brazil) with 1-minute interval after each application were performed. After that, the 2-step etch-and-rinse adhesive was applied in all etched surface (Adper Single Bond 2, 3M ESPE, St. Paul, MN, USA). Table 1 shows the composition of resin cements and adhesive system used in the cementation protocols.

The dentin of the post space was etched with 37% phosphoric acid (Dentsply, Pirassununga, SP, Brazil) for 15 seconds and rinsed with distilled water for 1 minute. After aspiration with tips (Cappillary tips; Ultradent, South Jordan, UT, USA), the space was gently dried with absorbent paper points to properly maintain the dentin moisture. The etch-and-rinse adhesive system (Adper Single Bond 2; 3M ESPE, ST. Paul, MN, USA) was actively applied in all dentin surface etched, in two steps of 15 seconds. Air-jet for 10 seconds was applied to evaporate the solvent and then the adhesive system was light-cured using a LED unit (Valo Cordless; Ultradent Products Inc., South Jordan, USA) with an irradiance of 1200 mW/cm², for 10 seconds. The LED unit was positioned perpendicular to the long axis of the root, as close as possible of the entry of the root canal, without touching the surface. During the irradiation, the LED was carefully held to avoid any movements capable of decreasing the light output.

The conventional dual resin cement (RelyX ARC; 3M ESPE, St. Paul, MN, USA) was handled and placed in the post space using a Rhein tip (Duflex SS White, Rio de

Janeiro, RJ, Brazil) to avoid the formation of voids and ensure complete fill. Immediately after, the fiber post was also coated with the resin cement and properly placed and stabilized in the post space. The rotatory instruments used in the post space preparation matched with the fiber post diameter, ensuring and standardizing its adaptation. Since the fiber post has a 20 mm length, 9 mm was above the surface of the root canal. Cervical excess material was removed and the assembly was light-cured for 20 seconds (Valo Cordless, Ultradent Products Inc., South Jordan, UT, USA). The irradiation was performed at the buccal, lingual, mesial, and distal surfaces of the root, positioning the LED unit perpendicular to each surface and above the fiber post. Then, the specimens were stored in distilled water for 24 hours at $37^{\circ}\text{C} \pm 1^{\circ}\text{C}$.

The roots were sectioned obtaining samples of the apical, middle and cervical thirds of the post space and were submitted to a push-out test as described by Ramos et al²³.

The maximum force to displace the set was given in Newtons (N) and then converted in megapascal (MPa) to evaluate the bond strength values, as described by Magro et al²².

Failure modes

To determine the failure modes, the specimens were evaluated using a stereomicroscope at 10x of magnification and classified according to Ramos et al⁶ in: type 1 (adhesive 1) – failure between the fiber post and the cement; type 2 (adhesive 2) – failure between the dentin and the cement; type 3 (cohesive) – failure within the cement; and type 4 (mixed) – several failures types combined.

Statistical analysis

Normality of the data was verified by Shapiro-Wilk test. For the incidence of residues data, Kruskal-Wallis and Dunn tests were employed. For the bond strength data, one-way ANOVA and Tukey post-hoc tests were used. Significance level of 5% was adopted to all the analyzes. The frequency of failure modes for each third of the post space was only descriptive.

RESULTS

Incidence of residues

At the cervical third of the post space, SH-PUI, CH-PUI and SH-ED-CI showed similar incidence of residues each other ($p > 0.05$), but lower than the other irrigations protocols (DW-CI, SH-CI and CH-CI) ($p < 0.05$). For DW-CI, SH-CI and CH-CI groups, the incidence of residues was also similar between each other ($p > 0.05$). At the middle

third, SH-ED-CI exhibited the lowest incidence ($p > 0.05$). Meanwhile, at the apical third, all the protocols showed similar incidence of residues ($p > 0.05$).

Table 2 displays the median, maximum and minimum values of the scores attributed for incidence of residues in the post-space dentin after the irrigation protocols. Similarly, Figure 1 shows the pattern observed for the incidence of residues for each group and thirds.

Bond strength

Regardless of the post-space third, DW-CI showed higher bond strength values (11.72, 11.81 and 11.60 MPa for cervical, middle and apical thirds respectively) than SH-CI, SH-PUI, CH-CI and CH-PUI ($p < 0.05$). No difference was observed between the bond strength values after irrigation with calcium or sodium hypochlorite, regardless of the method employed (CI or PUI) ($p > 0.05$) (Table 3).

Failure modes

Type 2 was the most frequent failure mode for SH-CI, SH-PUI, CH-CI and CH-PUI regardless of the post-space third ($\geq 60\%$). Differently, for the DW-CI groups, the failure mode more observed was the type 4 ($\geq 50\%$) (Figure 2).

DISCUSSION

Our results showed that SH-ED-CI, SH-PUI and CH-PUI exhibited the lowest incidence of residues at the cervical third, while for the middle third was SH-ED-CI. However, all the irrigation protocols showed similar incidence of residues at the apical third. Thus, the H_01 must be rejected. Regarding the push out bond strength, NaOCl and $\text{Ca}(\text{OCl})_2$, with or without PUI exhibited lower values than DW-CI, which lead us to also reject the H_02 .

DW-CI and SH-ED-CI were used as control groups to allow comparisons with the others groups. DW-CI was selected as the negative control protocol, since DW does not have cleaning potential to remove the residues over the post space dentin and does not exert effects on the bonding interface¹. On the other hand, SH-ED-CI was used as the positive control group, since intracanal irrigation with NaOCl and EDTA is widely recommended as the *gold-standard* protocol due to its potential to remove smear layer and depending on the resin cementation system may favor the bond strength of bonding interface^{2,3,10}.

Sample size was calculated based on a pilot study of the bond strength test which two specimens of each group and third were used. The values of standard deviation and sampling error were obtained and the minimum sample number to each

third was posteriorly defined. It is worth to mention that our sample size is in accordance with previous studies that used similar methodology^{5,6,23}.

SEM images of the dentin by resin-based replicas or low vacuum are used to evaluate the incidence of residues before irrigation protocols¹⁸. However, we use SEM images to only evaluated the incidence of residues of the dentin after the irrigation protocols, since our main goal was qualitatively comparing the incidence among the groups. Thus, the evaluation before the irrigation protocols was not required¹¹.

Several parameters have been reported to classify the incidence of residues over the root canal dentin surface²⁴. However, the residues formed after post space preparation are different from them formed after chemical-mechanical intracanal instrumentation. The post space preparation generates not only dentin debris but also residues from obturation materials^{5,15}. Based on these parameters, we use the scores described by Serafino et al¹⁵. which better represent the clinical scenario after post space preparation.

Push-out is one of the most used test to evaluate the bonding quality between the resin cementation system and the post space dentin substrate^{3,6}. To the axial displacement force be correctly distributed over the surface of the set fiber post/luting system, the crosshead diameter should occupy approximately 90% of the root canal diameter²⁵. For this reason, we avoid to compare the bond strength values at the different thirds since each third exhibits different diameters. In this study we avoid to use the same crosshead diameter for all the analyzes due to the risk of exert the displacement force only on the fiber post or dentin, which could result in bias in the bond strength and failure mode analyzes²⁶.

Failure mode interpretation allows to evaluate the regions of low bond strength between two surfaces²³. However, the classification in three failure types (adhesive, cohesive and mixed) does not allow the evaluation of the failure type that occur between the fiber post and the resin cementation system (adhesive 1)^{27,28}. Adhesive 1 failure mode was included in our study to ensure whether the bond strength values were not mistakenly obtained due to a deficiency in this interface bonding^{5,11,23}.

The profile of the incidence of residuals was different depending on the post-space third and the irrigation protocol. At the cervical third, SH-ED-CI and the protocols activated by PUI showed lower, but similar incidence between them, which corroborates with a previous report by Küçükekenci et al²⁹. PUI acts dislocating the residuals in a coronal direction as a result of the mechanical activation transmitted to

the solution by the oscillation of the ultrasonic tip. This oscillation provides cavitation effects and implosion of microbubbles that generates ultrasonic waves on the dentin and consequently removal of the smear layer³⁰. Thus, the lower incidence of residuals in the groups activated by PUI at the cervical third can be explained by the ultrasonic tip activation³¹.

At the middle third, SH-ED-CI exhibited the lowest incidence of residues. When the space post is prepared using rotatory instruments without cooling, heat is generated. This heat changes the physical state of filling materials which provides a precipitation of residuals over the intracanal dentin^{6,15}. Since the diameter of the middle third is smaller than the cervical third, the physical effects of ultrasonic activation tend to be less effective, which reduce the removal potential of residues and provide a greater incidence of covered dentin tubules¹⁸. This effect is crucial and even clearer at the apical third, since all the irrigation protocols showed similar incidence of residues, corroborating with Mirseifinejad et al¹⁰.

$\text{Ca}(\text{OCl})_2$ is more chemical stable and produces more hypochlorous acid than NaOCl . Besides, $\text{Ca}(\text{OCl})_2$ presents better biocompatibility due to the formation of residual calcium hydroxide^{12,14}. However, the cleaning potential and effects are similar between them¹³, regardless of the activation method used.

Our results showed that the bond strength of the resin cementation system was similar after the use of NaOCl or $\text{Ca}(\text{OCl})_2$ protocols, for all the post space thirds. Taking into account that the dentin was etched with phosphoric acid after the irrigation protocols, it may explain the similarity among these groups (Figure 1). Possibly, the phosphoric acid etching contributed to remove or alter the incidence of residues since it provides the partial removal of the smear layer over the dentin^{32,33}.

DW-CI showed higher bond strength than the other groups for all the thirds. The etching enhances the cleaning of the surface dentin which increased the hybridization in adhesive interface¹¹. Since the micromechanical retention is the main bonding mechanism of the adhesive to dentin^{11,23}, the higher bond strength for DW-CI may be explained by the fact that distilled water does not exert a direct effect on the bonding interface.

The chemical degradation process of NaOCl and $\text{Ca}(\text{OCl})_2$ generates sodium hydroxide and calcium hydroxide, respectively. However, these solutions also generate hypochlorous acid which posteriorly degrades into singlet oxygen. This highly-reactive oxygen molecule negatively affects the polymerization reaction and

consequently the bonding of etch-and-rinse adhesives to radicular dentin^{4,12}. Our results differ from those reported by Khoroushi et al⁹, however, they used a self-etching adhesive system after irrigation with 5.25% Ca(OCl)₂ which exhibits a different bonding mechanism based on chemical bonds between acidic monomers and calcium hydroxyapatite.

Moreover, the oxygen residues may fill spaces and reduce the adhesive diffusion into the dentin^{6,12}. This diffusion reduction can be confirmed by the higher incidence of adhesive 2 failure type for those groups that used NaOCl and Ca(OCl)₂, regardless of the third (Figure 2). In contrast, the mixed failure type was the most frequent for DW-Cl group.

Although we have provided relevant findings regarding novel irrigation protocols, some limitations must be considered. The post space was only aspirated after the irrigation and no final rinse with distilled water was performed, which could reduce the deleterious effects of irrigation solutions on the bonding interface, as described by Khoroush et al⁹. Moreover, push-out test was performed only 24 hours after the irrigation protocols. Thus, long-term evaluations (from 6 to 12 months) can exhibit different results due to the degradation of the bonding interface.

Within the limitations and the results of this study, further evaluations are crucial. Investigate different concentrations of NaOCl and Ca(OCl)₂, the use of self-adhesive cements or conventional cements associated with different self-etching adhesive systems, and perform long-term analyzes are strongly recommended to safely establish a viable clinical protocol.

CONCLUSION

Irrigation with 2,5% NaOCl and 2,5% Ca(OCl)₂, independently of the PUI activation, negatively impacted the bond strength of conventional dual resin cement (RelyX ARC) associated with 2-step etch-and-rinse adhesive (Single Bond 2) and maintained high incidence of residues in the post-space radicular dentin.

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The authors deny any conflicts of interest related to this study.

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Table and Figure Legends

Table 1. Composition of resin cement and adhesive system used in the cementation protocols.

Table 2. Median, maximum and minimum values of the attributed scores for the incidence of residues over the dentin surface in function of the irrigation protocols and the post space thirds.

Table 3. Mean, standard deviation and confidence intervals of the bond strength values (MPa) for the cementation system (Single Bond 2 and RelyX ARC) in function of the irrigation protocols and post space thirds.

Figure 1. Representative scanning electron microscopy images (500x) of irrigation protocols in dentin post space thirds. Scale: 20 μ m.

Figure 2. Representative images of the failure mode: (Type 1), adhesive 1, between pin and cementation system; (Type 2) adhesive 2, between dentin and cementation system; (Type 3) cohesive, inside the cementation system; (Type 4) mixed, combination of fracture pattern. Scale: 1024x.

Table 1. Composition of resin cement and adhesive system used in the cementation protocols.

Materials	Manufacturer	Composition
Adper Single Bond 2	3M ESPE, St. Paul, MN, USA	Dimethacrylate resins, hydroxyethylmethacrylate (HEMA), methacrylate-modified polyalkenoic acid copolymer (Vitrebond™ Copolymer), filler, ethanol, water, initiators
Relyx ARC	3M ESPE, St. Paul, MN, USA	Bis-GMA, TEGDMA, silanized zirconia/silica filler 68% functionalized dimethacrylate polymer, triphenyl antimony

Bis-GMA: bisphenol-glycidil methacrylate; TEGDMA: triethylene glycol dimethacrylate;

Table 2. Median, maximum and minimum values of the attributed scores for the incidence of residues over the dentin surface in function of the irrigation protocols and the post space thirds.

Post space third	Irrigation protocols					
	DW-CI	SH-ED-CI	SH-CI	SH-PUI	CH-CI	CH-PUI
Cervical	2 ^b	1 ^a	2 ^b	0.5 ^a	2 ^b	0.5 ^a
max-min	(2-2)	(1-0)	(2-2)	(1-0)	(2-2)	(1-0)
Middle	2 ^b	0.5 ^a	2 ^b	2 ^b	2 ^b	2 ^b
max-min	(2-2)	(1-0)	(2-2)	(2-1)	(2-2)	(2-1)
Apical	2 ^a	2 ^a	2 ^a	2 ^a	2 ^a	2 ^a
max-min	(2-2)	(2-1)	(2-2)	(2-1)	(2-2)	(2-1)

^{ab} Different lowercase letters within the same row mean statistically significant difference (Kruskal-Wallis and Dunn tests; $p < 0.05$). DW-CI: distilled water and conventional irrigation; SH-ED-CI: sodium hypochlorite associated with EDTA and conventional irrigation; SH-CI: sodium hypochlorite and conventional irrigation; SH-PUI: sodium hypochlorite and passive ultrasonic irrigation; CH-CI: calcium hypochlorite and conventional irrigation; CH-PUI: calcium hypochlorite and passive ultrasonic irrigation.

Table 3. Mean, standard deviation and confidence intervals of the bond strength values (MPa) for the cementation system (Single Bond 2 and RelyX ARC) in function of the irrigation protocols and post space thirds.

Post space thirds	Irrigation protocols					
	DW-CI	SH-ED-CI	SH-CI	SH-PUI	CH-CI	CH-PUI
Cervical	11.72 ^a	11.36 ^b	11.12 ^b	11.21 ^b	11.14 ^b	11.21 ^b
	(0.17)	(0.29)	(0.18)	(0.15)	(0.34)	(0.08)
	(11.63-11.81)	(11.19-11.53)	(11.01-11.23)	(11.12-11.30)	(10.94-11.34)	(11.16-11.26)
Middle	11.81 ^a	11.56 ^b	11.30 ^b	11.41 ^b	11.34 ^b	11.43 ^b
	(0.10)	(0.28)	(0.16)	(0.14)	(0.23)	(0.10)
	(11.76-11.86)	(11.41-11.71)	(11.20-11.40)	(11.34-11.47)	(11.21-11.47)	(11.37-11.48)
Apical	11.60 ^a	11.32 ^b	11.24 ^b	11.26 ^b	11.27 ^b	11.27 ^b
	(0.28)	(0.07)	(0.13)	(0.12)	(0.18)	(0.07)
	(11.48-11.72)	(11.29-11.35)	(11.16-11.32)	(11.21-11.35)	(11.15-11.29)	(11.16-11.27)

^{ab} Different lowercase letters within the same row mean statistically significant difference (one-way ANOVA and Tukey post-hoc test; $p < 0.05$). c: cervical third; m: middle third; a: apical third; DW-CI: distilled water and conventional irrigation; SH-ED-CI: sodium hypochlorite associated with EDTA and conventional irrigation; SH-CI: sodium hypochlorite and conventional irrigation; SH-PUI: sodium hypochlorite and passive ultrasonic irrigation; CH-CI: calcium hypochlorite and conventional irrigation; CH-PUI: calcium hypochlorite and passive ultrasonic irrigation.

3.2 Publicação 2*

Calcium hypochlorite to remove methylene blue after PDT improves the bond strength to post space dentin

Abstract

Background: Herein, we evaluated the effect of irrigation protocols in removing 0.01% methylene blue after PDT in post space dentin. Bond strength to dentin and failure mode after fiber post cementation with self-adhesive resin cement or glass ionomer cement was also evaluated.

Materials and Methods: Ninety bovine teeth were endodontically treated. Post space preparation and PDT mediated by 0.01% methylene blue and irradiated with diode laser (660 nm) were performed. Three irrigations protocols were tested to remove methylene blue by incidence of residues over dentin (n=10): distilled water (DW), 2.5% sodium hypochlorite (SH), and 6% calcium hypochlorite (CH). Other specimens were used to investigate the bond strength to post space dentin according to the irrigation protocol and the post cementation system used (RelyX U200 cement (RU) and GC Gold Label 1 glass ionomer cement (GC) in 6 groups (n=10): DW-RU, SH-RU, CH-RU, DW-GC, SH-GC e CH-GC.

Results: Regardless of the post space third, DW showed a lower incidence of residues than SH ($p < 0.05$), but similar to CH ($p > 0.05$). On the other hand, SH showed similar results to CH ($p > 0.05$). Regarding the bond strength to dentin, CH-RU and CH-GC showed higher values compared to the other groups ($p < 0.05$), but similar to each other ($p > 0.05$). No differences were observed between the other groups ($p > 0.05$). Cohesive was the most predominant failure.

Conclusions: 6% calcium hypochlorite to remove 0.01% methylene blue after PDT favors the bond strength of Relyx U200 and GC Gold Label.

Keywords: Fiber Post; Photodynamic Therapy; Bond strength; Methylene blue; Glass-ionomer cement; Self-adhesive cement

* Artigo elaborado seguindo as normas do periódico *Journal of Clinical and Experimental Dentistry*, para o qual o trabalho será submetido.

INTRODUCTION

During the post space preparation, the root canal may be contaminated with microorganisms from saliva (1). Photodynamic therapy (PDT) is a coadjuvant therapy with antimicrobial potential, reducing the microorganisms load inside the root canal (2). The antimicrobial activity occurs due to the interaction between a light source and a photosensitizer, such as 0.01% methylene blue, that promotes the formation and release of oxidative radicals, such as singlet oxygen, causing cell damage/death (3,4).

The use of dyes such as methylene blue can change the tooth color, compromising rehabilitation aesthetic procedures (5–8). To minimize these undesirable effects, several protocols have been recommended, mainly using oxidative solutions to clarify the tissue, such as sodium hypochlorite (NaOCl) at different concentrations (8,9). However, during the chemical dissociation of NaOCl there is the formation of sodium hydroxide and hypochlorous acid, that negatively affect the bonding interface with conventional cementation systems with etch-and-rinse adhesives (9,10). Considering these shortcomings, the use of 6% calcium hypochlorite solution ($\text{Ca}(\text{OCl})_2$), which also has the ability to clarify tissues, has been purposed (11). Although $\text{Ca}(\text{OCl})_2$ has chlorine in its composition, more chemical stability compared to sodium hypochlorite is observed, allowing a more gradual ionic dissociation which tends to minimize the impact on the adhesive interface with conventional resin cement (12–14).

To optimize clinical time and minimize errors during fiber post cementation, self-adhesive resin cement, and glass ionomers have been used over conventional resin cement (10,15). The adhesion of these cements occurs through bonds between the chemical components of the cementation system with the calcium of the hydroxyapatite of the dentinal substrate (15,16). On the other hand, calcium hypochlorite solution dissociates into calcium hydroxide, that precipitate on the dentin surface. However, the effects of the interaction between these residues and the cementation systems are still unknown (14).

This study evaluated the effects of removing 0.01% methylene blue after PDT in post space dentin using irrigation with distilled water, 2.5% sodium hypochlorite solution, or 6% calcium hypochlorite. Bond strength to dentin and failure mode after fiber post cementation with self-adhesive resin cement (Relyx U200) or glass ionomer cement (GC Gold Label 1 Luting & Lining Cement) at cervical, middle and apical thirds

of the post space was also evaluated. The null hypotheses tested were: H01 - There is no difference between the irrigation protocols in the incidence of residues on the dentin surface; H02 - There is no difference in bond strength and failure mode between the cementation systems.

MATERIAL AND METHODS

This study was approved by the Ethical Committee on Animal Use from the São Paulo State University, School of Dentistry, Araraquara (CEUA/FOAr-UNESP; registration number:19/2017).

Sample preparation

Ninety bovine conoid teeth, with similar root anatomy, were obtained. After selection, they were immersed in saline solution and kept under refrigeration at 4°C, until their use. The roots were transversally sectioned at 17 mm from the apex with a diamond disk coupled in a precision tissue cutting machine (Isomet 2000; Buehler Ltd., Lake Buff, IL, USA).

The root canals treatment was performed by a single operator, as described by Aranda-Garcia et al. (17). After cutting and vertical condensation of the endodontic filling, the coronal access was sealed with glass ionomer cement (Maxxion R; FGM, Joinville, SC, PR, Brazil). Then, the specimens were stored at 37°C and 100% humidity, for 7 days. After that, the provisional restoration was removed and the post space was prepared using Largo burs #1 and #2 (Dentsply Maillefer; Ballaigues, Jura-Nord Vaudois, Switzerland) and finished with a #2 bur (White Post DC; FGM, Joinville, SC, Brazil), under irrigation with distilled water, in an extension of 11 mm, from the cervical root surface. Then, the post space was irrigated with 5 mL of distilled water, aspirated and dried with absorbent paper points.

Photodynamic therapy

The post space was filled with 0.01% methylene blue solution (Chimiolux; DMC, São Carlos, SP, Brazil) and kept in the dark for 3 minutes by sealing the coronal access with an aluminum foil (Wyda; São José dos Campos, SP, Brazil). Subsequently, the post space was irradiated with a diode laser device (Therapy XT; DMC, São Carlos, SP, Brazil) under 660 nm wavelength coupled in a 300 µm diameter optical fiber. The device parameters were set in 100 mW of input and a light dose of 9 J (320 J/cm²).

The optical fiber was initially placed in the entire length of the post space followed by helical movements in apical-cervical directions with 3 mm of amplitude. An irradiation time of 90 seconds was used. After the irradiation had been completed, the post space was irrigated according to each protocol, aspirated with an endodontic tip (Capillary Tips; Ultradent, South Jordan, UT, USA), and dried with absorbent paper points.

Incidence of residues

Irrigation protocols

For this evaluation, thirty specimens were randomly assigned into 3 groups ($n = 10$), according to the irrigation protocol used to remove the photosensitizer solution:

DW (distilled water): irrigation with 5mL of distilled water using irrigator tips (Navitip 30G; Ultradent, South Jordan, UT, USA). The tips were placed at 10 mm inside the post space, in continuous movements from apical to cervical third;

SH (sodium hypochlorite): irrigation with 5mL of 2.5% sodium hypochlorite (Asfer Indústria Química, São Caetano do Sul, SP, Brazil) followed by aspiration (Capillary tips; Ultradent, South Jordan, UT, USA);

CH (calcium hypochlorite): similar to SH protocol, but using 6.0% calcium hypochlorite (Synth, São Paulo, SP, Brazil);

After performing all the irrigation protocols, the post space was aspirated and dried with absorbent paper points.

SEM analysis

After post space irrigation, longitudinal grooves in the bucco and lingual surfaces of the roots were made with a double face diamond-coated disc (KG Sorensen, São Paulo, SP, Brazil). The roots were then cleaved in buccolingual direction using a chisel (Golgran, São Paulo, SP, Brazil). To standardize the evaluation, only the distal sections of the roots were analyzed.

Three delimitations in the buccal surface of the post space were performed at 3, 6 and 9 mm from the cervical surface to identify the cervical, middle and apical third respectively. Afterward, the specimens were stored at $37^{\circ}\text{C} \pm 1^{\circ}\text{C}$ for 5 days and dried at a desiccator with silica gel for 2 days. The specimens were then individually placed on metallic stubs and coated in gold-palladium (Bal-Tec, Balzers, Liechtenstein), at 20 mA for 180 seconds. Four areas of each third of the post space were analyzed and a representative image was photographed at 500x of magnification (LEO 435VP, Carl

Zeiss Microscopy Ltd, Cambridge, UK) in 10 kV. All images were photographed by a single, independent, and blind operator.

The incidence of residues in each third of the post space dentin surface was evaluated as previously described by Serafino et al.(18). The scores were graded between 0 and 2: score 0 – all dentin tubules were open and no debris or sealer remnants covering the opening of the tubules were verified; score 1 – most of the dentin tubules were open and some debris or sealer remnants covering the opening of the tubules were verified; score 2 – most of the dentin tubules were covered and a great amount of debris or sealer remnant were verified.

Push-out bond strength

Experimental groups

Sixty specimens were randomly allocated into 6 groups (n=10) according to the irrigation protocol used to remove the photosensitizer and the cementation system used (Table 1):

DW-RU (distilled water and Relyx U200): Initially, the surface of the fiber post (#2DC Whitepost; FGM, Joinville, SC, Brazil) was cleaned with 95% ethyl alcohol solution (Rinse-N-Dry; Vista Dental, Racine, WI, USA), etched with 37% phosphoric acid (Condac; FGM, Joinville, SC, Brazil) for 60 seconds, and rinsed with distilled water. Then, two layers of silane were applied (Prosil; FGM, Joinville, SC, Brazil) and dried with a gentle air-jet, according to the manufacturer's recommendations. After the irrigation protocol with DW as previously described, the self-adhesive resin cement (Relyx U200, color A2; 3M ESPE, St. Paul, MN, USA), was placed inside the post space using a customized automix insertion tip. After, the fiber post was properly positioned and the excess of material was removed. The set was light-cured with LED unit (Valo Cordless, Ultradent Products Inc., South Jordan, UT, USA), with an irradiance of 1000 mW/cm² for 40 seconds on each surface of the root (buccal, lingual, distal and mesial). Then, the specimens were stored in distilled water for 24 hours at 37°C ± 1°C;

SH-RU (sodium hypochlorite and Relyx U200): Similar to that described in DW-RU, but the irrigation protocol was performed with 2.5% sodium hypochlorite solution (Asfer, São Caetano do Sul, SP, Brazil);

CH-RU (calcium hypochlorite and Relyx U200): Similar to that described in DW-RU, but the irrigation protocol was performed with a 6% calcium hypochlorite solution (Sauer, São Paulo, SP, Brazil);

DW-GC (distilled water and GC Gold 1 Label Luting & Lining): The surface of the fiber post (#2DC, Whitepost; FGM, Joinville, SC, Brazil) was cleaned with 95% ethyl alcohol solution (Rinse-N-Dry; Vista Dental, Racine, WI, USA), etched with 37% phosphoric acid (Condac; FGM, Joinville, SC, BR) for 60 seconds, and washed with distilled water. After the post space irrigation with DW as previously described, the glass ionomer cement (GC Gold Label Luting & Lining) was properly handled, according to the manufacturer's recommendations, and inserted into the post space using a Lentulo spiral. Then, the fiber post was properly positioned.

SH-GC (sodium hypochlorite and GC Gold Label 1 Luting & Lining): Similar to that described in DW-GC, but the irrigation protocol was performed with 2.5% sodium hypochlorite solution (Asfer, São Caetano do Sul, SP, Brazil);

CH-GC (calcium hypochlorite and GC Gold Label Luting & Lining): Similar to that described in DW-GC, but the irrigation protocol was performed with a 6% calcium hypochlorite solution (Sauer, São Paulo, SP, Brazil).

Push-out test

The roots were vertically included in a polyvinyl chloride matrix. These matrices were filled with polyester resin (Maxi Rubber, Diadema, SP, Brazil) up to 16 mm of height to leave 1 mm of the cervical third out of the inclusion (19). After 24 hours, the specimens were removed from the matrices and transversally sectioned with a diamond saw coupled in a precision water-cooled cutter machine (Isomet 1000, Buehler Ltd, Lake Bluff, IL, USA). Three sections with $2 \text{ mm} \pm 1 \text{ mm}$ thickness from the apical, middle and cervical thirds of the post space were obtained. Cervical, middle and apical sections were obtained from 1, 5 and 8 mm from the root cervical surface, respectively. Irregularities were removed using 1200-grit silicon carbide sandpapers (Norton, São Paulo, SP, Brazil).

Push-out test was performed using an electromechanical test machine (EMIC, São José dos Pinhais, PR, Brazil) with 5 kN load-cell at 0.5 mm/min speed. To displace the set fiber-post/luting-system, a specific notched crosshead for axial displacement was used for each third: cervical 1.2 mm, middle 0.9 mm, and apical 0.5 mm. The maximum force to displace the set was given in Newtons (N) and then converted in

megapascal (MPa) to evaluate the bond strength values, as described by Magro et al. (20).

Failure mode analysis

After push-out bond strength tests, the specimens were regularized using #600 and #1200 silicon carbide sandpapers (Norton, Lorena, SP, BR). Then, rinsing with distilled water was performed and the cervical surface was polished using aluminum oxide (30µm-grit size; Arotec, São Paulo, SP, BR). After that, the specimens were submitted to ultrasonic bath in distilled water (Cristófoli, Campo Mourão, PR, USA) for 10 minutes. Then, the specimens were dried using air spray and fixed on a glass slide in horizontal position. To determine the failure modes, a stereomicroscope was used at 10x of magnification and classified according to Ramos et al. (19) in: type 1 (adhesive 1) - failure between the fiber post and the cement; type 2 (adhesive 2) - failure between the dentin and the cement; type 3 (cohesive) - failure within the cement; and type 4 (mixed) - several failures types combined.

Statistical analysis

Data from the incidence of residues were submitted to Kruskal Wallis and Dunn test. Data from push-out bond strength were submitted to two-way ANOVA and Tukey post-hoc tests. A significance level of 5% was adopted to all the analyzes using the SPSS statistic software (IBM SPSS, New York, NY, USA). Images of failure mode were qualitatively analyzed.

RESULTS

Incidence of residues

Table 2 shows the scores attributed to the incidence of residues on the dentin surface, as a function of the irrigation protocols used to remove methylene blue after PDT and the third of the post space. Regardless of the third, DW showed lower incidence of residues than SH protocol ($p < 0.05$), but similar to CH protocol ($p > 0.05$). On the other hand, SH protocol exhibited similar incidence of residues to CH protocol ($p > 0.05$). Figure 1 shows the representative images of the incidence of residues on the dentin surface, as a function of the irrigation protocol and the third of the post space.

Push-out bond strength

Table 3 shows the bond strength values (in MPa) in the dentin of the post space thirds as a function of the irrigation protocols and fiber post cementation system used. Regardless of the post space third, CH-RU and CH-GC showed higher bond strength values compared to the other groups ($p < 0.05$), but they were similar to each other ($p > 0.05$). No difference between the other groups was observed ($p > 0.05$).

Failure mode incidence

Figure 2 shows the representative images of failure modes after push-out test. Cohesive failure was the most incident failure mode regardless of the type of irrigation protocol or fiber post cementation system used (Figure 3).

DISCUSSION

Removal of 0.01% methylene blue after PDT with calcium hypochlorite solution causes the precipitation of residues on the dentin surface of the post space. However, these residues do not appear to interfere with the fiber post cementation using self-adhesive resin cement (Relyx U200) or conventional glass ionomer cement (GC Gold Label 1 Luting & Lining), since the bond strength values of calcium hypochlorite group were higher than distilled water and 2.5% sodium hypochlorite. Therefore, the null hypotheses H01 and H02 must be rejected.

PDT is an antimicrobial approach to control the contamination of the root canals during the post space (19). Methylene blue is one of the most used photosensitizers, but it can change the color of the tooth structure (8). Thus, irrigation solutions with the ability to clarify tissues have been used, especially sodium hypochlorite, to prevent and/or overcome this limitation (9).

In our study, the incidence of residues over the post space dentin surface was assessed by SEM, directly on the specimen under evaluation (18,21,22). Several parameters were used to classify the presence of debris and/or smear layer in root dentin after the chemical-mechanical preparation of root canals (20,22). However, the characteristic of the residues formed after post space preparation tends to be different, due to the presence of residues from the endodontic filling and/or debris derived from the prosthetic preparation itself (16,21). Thus, we chosen to evaluate the incidence of residues based on the parameters described by Serafino et al. (18), who described the characteristic of the debris formed especially on the post space dentin.

Distilled water was used as a control solution, since it has no clarifying effects and does not affect the bonding interface of fiber post cementation systems (1). Sodium hypochlorite exhibit adequate antimicrobial activity, but persistent residues on the dentin of the post space can be observed (Figure 1; SH group), mainly in apical third due to the hindered access to this region (23,24). Calcium hypochlorite is an alternative irrigant with similar chemical and antimicrobial properties compared to NaOCl (25). Despite persistent residues were also observed in CH group regardless of the post space third (Figure 1), the characteristics of the debris are smaller and less diffused into the root dentin (11).

The effects of these irrigation protocols on the bonding interface between the dentin and the cementation system are crucial to select the most adequate material (26,27). In our study, the push-out bond strength test was used to assess the interference of sodium or calcium hypochlorite on the bonding interface with the cementation systems (9,23). The diameters of the crossheads were different for each post space third to obtain an adequate contact with the fiber post assembly and cementation system (28). Thus, we only performed bond strength comparisons between within the same thirds as a function of the different groups (16,27).

Regardless of the third, the removal of 0.01% methylene blue with 6% calcium hypochlorite provided greater bond strength to root dentin, independently of the cementation system used in comparison with other irrigation protocols (Table 2). Sodium hydroxide and calcium hydroxide are by-products derived from the ionic dissociation of sodium hypochlorite and calcium hypochlorite, respectively (11,17). After irrigation of the root canals, these by-products tend to precipitate into the root dentin (17,20). Considering that the adhesion mechanism of self-adhesive resin cements and conventional glass ionomer involves the chemical reaction with the calcium from the hydroxyapatite of the dentinal substrate, it is possible that a synergism of action between the precipitated residues of calcium hypochlorite with these cementation systems occurred, increasing the bond strength values (13,29). On the other hand, our results diverge from those shown by Seballos et al. (30), but the concentration of calcium hypochlorite used in our study was 6%, which crucially tends to provide greater precipitation of calcium hydroxide, compared to other solutions with lower concentrations. The bond strength was evaluated 24 hours after fiber post cementation. Thus, it must be considered that the integrity and longevity of the

adhesive interface has a direct relationship with the time factor. In this way, our results must be carefully evaluated with a short-term response.

The failure mode analysis collaborates in the interpretation of the results obtained in the push-out test (19). Cohesive failure was the most incident failure (Figure 3), regardless of the irrigation protocol, which suggests that cementation systems showed suitable adhesion due to chemical reactions with the dentin substrate, as well as, the oxidative radicals released from hypochlorite solutions do not interfere on this type of adhesive interface (9,24).

Although this study brings relevant clinical information regarding new irrigation protocols for the removal of methylene blue at 0.01% from the post space after PDT, some considerations must be addressed. Long-term evaluations may demonstrate different results due to the degradation of the adhesive interface. We encourage further studies that complement the irrigation protocol to remove photosensitizer agents used in PDT, such as final irrigation with distilled water after sodium or calcium hypochlorite, as well as analyzes with other fiber post cementation systems.

CONCLUSION

The use of 6% calcium hypochlorite to remove 0.01% methylene blue after PDT favors the bond strength of self-adhesive resin cement (RelyX U200) and conventional glass ionomer cement (GC Gold Label 1) to post space dentin.

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TABLES

Table 1. Composition of materials used in the cementation protocols.

Materials	Manufacturer	Composition
Relyx U200	3M ESPE, St Louis, MN, USA	Base paste: Silane treated glass filler, 2-propenoic acid, 2-methyl 1,10-[1-(hydroxymethyl)-1,2 ethanediyl] ester, triethylene dimethacrylate, sodium persulfate and per-3,5,5-trimethylhexanoate t-butyl. Catalyst paste: Silanated filler, dimethacrylate, silane treated filler, sodium p-toluenesulfonate, 1-benzyl-5-phenyl-baric acid, calcium salts, 1,12 dodecane dimethacrylate, calcium hydroxide, and titanium dioxide.
GC Gold Label 1 Luting & Lining	CG America Inc, Alsip, USA	Distilled water (CAS 7732-18-5) 50-55% Polyacrylic acid (CAS 9003-01-4) 30-40%
Silane Prosil	FGM, Joinville, SC, BR	3-Metacriloxypropiltrimetoxisilano, ethanol, and water

Table 2. Median and interquartile range (1Q-3Q) of scores attributed to the incidence of residues on the dentin surface in function of the irrigation protocols and the post space thirds.

Groups	Post space thirds		
	Cervical	Middle	Apical
DW	1 (0-1) ^a	1 (0-2) ^a	1 (0-2) ^a
SH	2 (1-2) ^b	2 (2-2) ^b	2 (2-2) ^b
CH	1 (1-2) ^{ab}	1 (1-2) ^{ab}	1.5 (1-2) ^{ab}

^{ab} Different letters within the same columns show statistical difference ($P < 0.05$). DW: distilled water irrigation protocol; SH: 2.5% sodium hypochlorite irrigation protocol; CH: 6% calcium hypochlorite irrigation protocol.

Table 3. Mean and standard deviation of the push-out bond strength values (MPa) in function of the protocol irrigation and the resin cement system, in post space thirds.

Post space thirds	DW-RU	SH-RU	CH-RU	DW-GC	SH-GC	CH-GC
C	9.03 ^b (0.47)	9.01 ^b (0.49)	11.09 ^a (0.61)	9.63 ^b (0.48)	9.32 ^b (0.57)	11.14 ^a (0.39)
M	9.05 ^b (0.51)	8.85 ^b (0.41)	10.58 ^a (0.52)	9.39 ^b (0.41)	9.01 ^b (0.43)	10.75 ^a (0.56)
A	7.86 ^b (0.54)	7.55 ^b (0.51)	9.68 ^a (0.58)	7.99 ^b (0.54)	7.54 ^b (0.51)	9.71 ^a (0.68)

^{ab} Different letters within the same line show statistical difference ($P < 0.05$). C: cervical third; M: middle third; A: apical third; DW-RU: distilled water irrigation protocol and Relyx U200; SH-RU: 2.5% sodium hypochlorite irrigation protocol and Relyx U200; CH-RU: 6% calcium hypochlorite irrigation protocol and Relyx U200; DW-GC: distilled water irrigation protocol and GC Gold Label 1 Luting & Lining; SH-GC: 2.5% sodium hypochlorite irrigation protocol and GC Gold Label 1 Luting & Lining; CH-GC: 6% calcium hypochlorite irrigation protocol and GC Gold Label 1 Luting & Lining.

Figure legends

Figure 1. Representative scanning electron microscopy images (500x) of irrigation protocols to 0.01% methylene blue removal, in dentin surface post space thirds. DW, distilled water; SH, 2.5% sodium hypochlorite; CH, 6% calcium hypochlorite. Scale: 100µm.

Figure 2. Representative of the failure modes images characteristics. A. type 1 (adhesive): between the fiber post and cement; B. type 2 (adhesive): between dentin and cement; C. type 3 (cohesive) within the cement; D. type 4 (mixed) when both types of failure were combined. Magnification: 10x

Figure 3. Incidence of the adhesive failure mode according to the groups evaluated. DW-RU, distilled water irrigation protocol and Relyx U200; SH-RU, 2.5% sodium hypochlorite irrigation protocol and Relyx U200; CH-RU, 6% calcium hypochlorite irrigation protocol and Relyx U200; DW-GC, distilled water irrigation protocol and GC Gold Label 1 Luting & Lining; SH-GC, 2.5% sodium hypochlorite irrigation protocol and GC Gold Label 1 Luting & Lining; CH-GC, 6% calcium hypochlorite irrigation protocol and GC Gold Label 1 Luting & Lining.

4 DISCUSSÃO

Nossos resultados mostraram que SH-ED-CI, SH-PUI e CH-PUI apresentaram a menor incidência de resíduos no terço cervical, enquanto para o terço médio foi SH-ED-CI. No entanto, todos os protocolos de irrigação apresentaram incidência semelhante de resíduos no terço apical. Assim, o H01 deve ser rejeitado. Em relação à resistência de união, NaOCl e Ca(OCl)₂, com ou sem PUI, apresentaram valores mais baixos do que o DW-CI, o que nos levou a também rejeitar o H02.

A remoção de 0,01% azul de metileno após PDT com solução de hipoclorito de cálcio a 6% causa a precipitação de resíduos na superfície de dentina do espaço para pino. No entanto, esses resíduos não parecem interferir com a cimentação dos pinos de fibra usando um cimento resinoso autoadesivo (Relyx U200) ou um cimento de ionômero de vidro convencional (GC Gold Label 1 Luting & Lining), uma vez que os valores de resistência de união do grupo de hipoclorito de cálcio foram maiores do que a água destilada e 2,5% hipoclorito de sódio. Portanto, as hipóteses nulas H01 e H02 devem ser rejeitadas.

5 CONCLUSÃO

A irrigação com 2,5% de NaOCl e 2,5% Ca(OCl)₂, independentemente da ativação com PUI, impactou negativamente sobre a resistência de união do cimento resinoso convencional dual (RelyX ARC) associado ao sistema adesivo condiciona e lava de 2 passos (Single Bond 2) e manteve alta incidência de resíduos na dentina radicular do espaço preparado para pino.

Por outro lado, o uso do hipoclorito de cálcio a 6% para remover 0,01% de azul de metileno após o PDT favoreceu a resistência de união do cimento resinoso autoadesivo (RelyX U200) e do cimento de ionômero de vidro convencional (GC Gold Label 1) à dentina intrarradicular do espaço preparado para pino.

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* 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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ANEXO A – Submissão para o periódico Operative Dentistry

Operative Dentistry

Bonding and cleaning effects of irrigation protocols using calcium hypochlorite on the post-space radicular dentin
--Manuscript Draft--

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Abstract:	This study evaluated the effect of 2.5% sodium hypochlorite (SH) or calcium hypochlorite (CH) submitted to passive ultrasonic irrigation (PUI) or conventional irrigation (CI) on the incidence of residues and the bond strength of the cementation system to post space radicular dentin. Distilled water (DW) and 2.5% SH followed by 17% EDTA (SH-ED) were used as control groups. Cervical, middle and apical thirds of the post space were evaluated. 120 bovine incisors were endodontically treated and post space preparation was performed. The specimens were randomly assigned to 6 groups, according to the solution and irrigation method: DW-CI, SH-ED-CI, SH-CI, SH-PUI, CH-CI, and CH-PUI. Incidence of residues (n = 10) over the dentin was evaluated by scores using SEM images. Other specimens were irrigated as previously described and the post cementation was immediately performed using a conventional dual resin cement and a 2-step etch-and-rinse adhesive system. Push-out and failure mode were performed for bonding evaluation. Kruskal-Wallis and Dunn test for incidence of residues data and one-way ANOVA and Tukey tests for bond strength data were used at a significance level of 5%. The protocols that showed lower incidence of residues were: SH-ED-CI, SH-PUI and CH-PUI for cervical third and SH-ED-CI for middle third (p < 0.05). At apical third, the protocols were similar each other (p > 0.05). Bond strength values were higher after irrigation with DW-CI for all thirds (p < 0.05). 2.5% sodium or calcium hypochlorite negatively impacted the adhesion interface and exhibit greater incidence of residues over the post space radicular dentin.

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(Direitos de publicação reservados ao autor)

Araraquara, 05 de setembro de 2022.

Giovanna Righetti Bravo