UNESP - Universidade Estadual Paulista“Júlio de Mesquita Filho”

Faculdade de Odontologia de Araraquara

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Efeitos do condicionamento do substrato dentinário sobre a interface de adesão com os sistemas de cimentação de pinos de fibra

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Tese apresentada à Universidade Estadual Paulista (Unesp), Faculdade de Odontologia de Araraquara para obtenção do título de Doutor em nome do Programa Ciências Odontológica, na Área de Dentística Restauradora.

Orientador: Prof. Dr.Milton Carlos Kuga

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Comissão julgadora

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DADOS CURRICULARES

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Dedico

Àquele de ombros largos, por onde sempre me apoiei:

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RESUMO

No Artigo 1, avaliamos os efeitos do glutaraldeído e do ácido tânico, como agentes promotores do *crosslink* da matriz de colágeno, sobre a interface de adesão entre a dentina do espaço intrarradicular preparado para núcleo de fibra e os sistemas de cimentação condiciona-e-lava e autocondicionante. As amostras foram mantidas imersas em saliva artificial por 48 horas e 12 meses. Em seguida, as secções foram submetidas ao ensaio mecânico de *push-out*, teste de padrão de fratura e penetrabilidade dentinária em microscopia de fluorescência confocal. Já no Artigo 2, foi avaliado o efeito dos protocolos de irrigação na remoção de azul de metileno 0,01% após a PDT na dentina radicular preparada para pino. A resistência de união à dentina e o modo de falha adesiva após a cimentação do pino de fibra com cimento resinoso autoadesivo e cimento de ionômero de vidro também foram avaliados. Três protocolos de irrigação foram testados para remoção do azul de metileno por incidência de resíduos sobre a dentina: água destilada, hipoclorito de sódio 2,5% e hipoclorito de cálcio 6%. No Artigo 3, avaliamos o efeito dos protocolos de irrigação na dentina radicular do espaço protético, com ácido peracético a 1%, Smear Clear ou solução QMix em comparação com hipoclorito de sódio a 2,5% na resistência de união e penetrabilidade da dentina usando um sistema de cimentação autoadesivo.

Palavras-chave: Dentina. Endodontia. Adesividade.

Morais JMP. Effects of dentin substrate conditioning on the bonding interface with fiber post cementation systems [tese de doutorado]. Araraquara: Faculdade de Odontologia da UNESP; 2022.

ABSTRACT

In Article 1, we evaluated the effects of glutaraldehyde and tannic acid, as agents that promote collagen matrix cross-linkers, on the adhesion interface between the dentin of the intraradicular post space dentin and the cementation with etch and rinse and self-adhesive. The samples were kept immersed in artificial saliva for 48 hours and 12 months. Then, the sections were submitted to the mechanical push-out test, failure mode test and dentin penetrability in confocal fluorescence microscopy. In Article 2, 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. Three irrigation protocols were tested to remove by 0.01% methylene blue by incidence of residues over dentin: distilled water, 2.5% sodium hypochlorite and 6% calcium hypochlorite. In Article 3, we evaluated the effects of surface treatments on post space root dentin with 1% peracetic acid, Smear Clear or QMix solution compared to 2.5% sodium hypochlorite. on bond strength and dentin penetrability using a self-adhesive cementation system.

Keywords: Dentin. Endodontics. Adhesiveness.

SUMÁRIO

1 INTRODUÇÃO

Uma das principais causas das falhas adesivas é adegradação da camada híbrida pela excessiva presença de água no local (degradação hidrolítica) e/ou a destruição da matriz de colágeno por proteases presentes na dentina[1,2].

A hidratação do substrato dentinário é importante para evitar a aproximação das moléculas do colágeno, a qual ocasionaria o estabelecimento de novas pontes de hidrogênio entre as cadeias peptídicas adjacentes, ocasionando o colapso desta matriz, que ocasionaria a redução do espaço interfibrilar dificultando a infiltração do sistema adesivo e, consequentemente a formação de uma camada híbrida imperfeita[3,4].

Por outro lado, a presença excessiva de água favorece a atividade das metaloproteinases (MMPs), principalmente as MMP-2, -3, -8, -9 e -20, e das catepsinas cisteínas, que ocasionam a destruição precoce da matriz de colágeno, por adição de uma molécula de água na cadeia polipeptidea[5-7].

Portanto, a longevidade da camada hibrida e a integridade da interface adesiva entre o substrato dentinário e o sistema adesivo, estão diretamente relacionados à resistência intrínseca de seus constituintes ao processo de degradação[8].Como o colágeno é extremamente sensível à degradação proteolítica e/ou hidrolítica, algumas substâncias têm sido recomendadas para reduzir esta degradação e também para incrementar o *crosslink* (entrelaçamento) entre as fibras colágenas[9].

O crosslink da matriz de colágeno, previamente ao tratamento endodôntico ou restaurações estéticas poderia proporcionar diversas vantagens, tais como: resistência da degradação da interface adesiva; prevenir a recolonização bacteriana na interface de adesão e incrementar as propriedades mecânicas da matriz de colágeno[10].

Na vertente de sanificação do sistema de canal radicular, durante o preparo do espaço para pino de fibra pode ocorrer a contaminação do mesmo com microrganismos provenientes da saliva. A terapia fotodinâmica também pode contribuir para o controle microbiano e antissepsia do local. A atividade antimicrobiana ocorre devido a interação entre o laser diodo de baixa potência com um fotossensibilizador, tal como o azul de metileno a 0.01%, que promove a formação e liberação de radicais oxidativos, tais como o oxigênio singlet[11].

O azul de metileno tem sido utilizado como alvo para microrganismos da microbiota endodôntica. Entretanto, ele pode ocasionar a alteração da coloração da estrutura dental e comprometer a estética do dente envolvido[12-14]. Com o objetivo de minimizar estes efeitos indesejáveis, diversos protocolos foram preconizados, principalmente utilizando soluções oxidantes com poder clarificante, tal como o hipoclorito de sódio em diversas concentrações[15].

Como possibilidades menos onerosas e que não comprometam a estética dental, contudo proporcionem adesão satisfatória dos pinos à dentina radicular as soluções quelantes têm sido uma alternativa[16]. Estas, desempenham um papel importante na remoção de detritos e da smear layer, dentre elas, o QMix e o SmearClear associam propriedades quelantes do etilenodiaminotetraacético ácido (EDTA) [17,18].

Como alternativa, o ácido peracético (AP) tem se mostrado promissor para dissolver a smear layer e desinfetar o sistema de canais radiculares[18]. Ele se decompõe em subprodutos seguros e oxigênio, reduzindo a formação de smear layer de forma tão eficaz quanto o EDTA[19].

Apesar destas observações, os efeitos químicos destes agentes não tem sido adequadamente esclarecida[20]. Isto se torna mais crítico, quando se pensa nadentina do espaço preparado para pino de fibra. Sendo assim, ainda faltam informações de como seria o comportamento dassas substâncias sobre a dentina do canal radicular preparada para pino de fibra.

2 PROPOSIÇÃO

Publicação 1: Avaliar o efeito dos protocolos de irrigação com os crosslinkers glutaraldeído e ácido tânico, sobre o tratamento da dentina radicular do espaço preparado para pino de fibra.

Publicação 2: Avaliar o efeito dos protocolos de irrigação com hipoclorito de cálcio 6% ou sódio 2,5% , na remoção de azul de metileno a 0,01%, após PDT na dentina do espaço protético

Publicação 3: Avaliar os efeitos dos tratamentos de superfície na dentina radicular do espaço para pino, com ácido peracético a 1%, Smear Clear ou solução Qmix em comparação com o hipoclorito de sódio a 2,5%.

3 PUBLICAÇÕES

3.1 PUBLICAÇÃO 1

Efeito de crosslinkers sobre a interface de adesão entre o adesivo convencional*

* Artigo redigido de acordo com as normas da Operative Dentistry.

RESUMO

O objetivo do estudo foi avaliar os efeitos do condicionamento do substrato dentinário com o ácido tânico (AT) ou glutaraldeído (GL) em comparação a água destilada (AD), sobre a resistência de união e padrão de falha adesiva na interface de adesão com o adesivo etch-and-rinse , nos terços cervical, médio e apical do espaço para pino, após 48h e 1 ano. Sessenta raízes de incisivos bovinos foram tratadas endodonticamente e realizado o preparo do espaço para pino. As raízes foram distribuídas em três grupos, de acordo com o protocolo de condicionamento da dentina com AT, GL ou AD (n=20) e realizada a cimentação do pino de fibra com adesivo etch-and-rinse e cimento resinoso convencional. 48 horas (n=30) e 1 ano (n=30), espécimes dos terços cervical, médio e apical do espaço para pino foram submetidos a avaliação da resistência de união do sistema de cimentação, em máquina de ensaio mecânico. A falha adesiva foi avaliada por estereomicroscopia, Em 48 horas, AT e GL proporcionaram similares valores de resistência de união, independentemente do terço do espaço para pino ($p>0.05$), mas maiores que AD ($p<0.05$). Após 1 ano, GL demonstrou o maior valor de resistência de união ($p<0.05$), mas não houve diferença entre AT e AD independentemente do terço avaliado ($p>0.05$). A solução de ácido tânico a 5% não proporciona maior longevidade da interface adesiva entre a dentina do espaço para pino e o sistema de cimentação com adesivo condiciona-e-lava e cimento resinoso convencional.

Palavras-chave: Dentina. Endodontia. Adesivos Dentários.

1 INTRODUÇÃO

O deslocamento axial de pino de fibra após a conclusão do tratamento restaurador estético é um indesejável acontecimento clínico (Lorenzetti et al, 2019). Diversos fatores podem interferir negativamente sobre a interface de adesão entre a dentina radicular e o sistema de cimentação, tais como presença de resíduos, tipo de adesivo e cimento resinoso utilizado, bem como o protocolo de irrigação do espaço para pino (Ramos et al., 2018; Belizário et l., 2019; Alencar et al., 2021).

Entretanto, a degradação das fibras colágenas por metaloproteinases e catepsinas podem comprometer a longevidade da interface adesiva após o uso dos adesivos condiciona-e-lava (Malacarne et al, 2006 Seseogullari-Dirihan et al, 2016.). Para tanto, algumas substâncias condicionadoras do substrato dentinário tem sido recomendadas para uso prévio à cimentação dos pinos de fibra, no intuito de minimizar a degradação da matriz de colágeno (Carrilho et al., 2007).

Por outro lado, dependendo das características químicas dos adesivos e/ou do cimento resinoso utilizado para a cimentação dos pinos de fibra, somente o uso de inibidores ou atenuantes das ações das metaloproteinases pode não ser eficazes na manutenção da longevidade da camada híbrida (Pahsley et al., 2003; Hashimoto et al., 2004). Com o propósito de promover uma união mais coesa entre as fibras colágenas da dentina, também tem sido recomendado de condicionadores teciduais que provem o *crosslink*, ou seja, o entrelaçamento destas fibras, tais como o glutaraldeído e os flavanóides, tais como o tanino (Citta et al., 2021; Khalid et al., 2020; Alonso et al., 2018).

O gluraldeído é uma das substâncias utilizadas em produtos odontológicos para promover o *crosslink* das fibras colágenas e dessensibilização dentinária (Khalid et al, 2020). Porém, devido a sua toxidade é questionável o seu uso roteineiro durante os procedimentos odontológicos (Lee et al., 2017). Com o propósito de encontraramos substâncias com potencial antioxidante e indutor de *crosslink* na dentina do espaço para pino, os flavanóides são uma alternativa, tais como os derivados do ácido tânico (Polassi et al., 2021). Entretanto inexistem estudos que comprovem esta hipótese.

Portanto, o objetivo do presente estudo foi avaliar os efeitos dos protocolos de condicionamento do substrato dentinário com a solução de tanino a 5% ou glutaraldeído a 5%, em comparação a água destilada, sobre a resistência de união e padrão de falha adesiva na interface de adesão com o sistema de cimentação com adesivo condiciona-e-lava (Adper Scotchbond Multipurpose) e cimento resinoso convencional dual (Relyx ARC), nos terços cervical, médio e apical do espaço para pino, nos períodos de controle de 48 horas e 1 ano. A hipótese H_0 refere a inexistência de efeitos e a hipótese H_1 refere a existência de efeitos dos protocolos de condicionamento do substrato dentinário sobre a interface de adesão com o sistema de cimentação, nos períodos de 48 horas e 1 ano de controle.

2. Material e métodos

O presente estudo foi aprovado pelo CEUA/FOAr-UNESP (número de registro: 41/2017).

2.1. Preparo das amostras

Sessenta dentes conóides anteriores bovinos, com anatomia radicular semelhante, foram obtidos e previamente selecionados, imersos em solução salina e mantidos sob refrigeração a 4ºC, até o momento de uso. As raízes foram transversalmente seccionadas com disco diamantado adaptado em máquina de corte de tecidos (Isomet 2000; Buehler Ltd., Lake Buff, IL, USA) e padronizadas com comprimento de 18 mm a partir do ápice radicular.

O tratamento dos canais radiculares foi realizado conforme descrito por Aranda-Garcia et al. (2013). Após o corte cervical da obturação do canal radicular, o acesso cervical foi restaurado provisoriamente com cimento de ionômero de vidro (Maxxion R; FGM, Joinville, SC, PR, BR). Os espécimes foram armazenados a 37ºC, a 100% de umidade, por 7 dias. Após a remoção do ionômero de vidro, o espaço para pino foi preparado com brocas de Largo #1 e #2 (Dentsply Maillefer; Ballaigues, Jura-Nord Vaudois, Switzerland) e complementada com a broca #2 (White Post DC; FGM, Joinville, SC, BR), no comprimento de 15 mm, a partir da face cervical radicular. Em seguida, o espaço para pino foi irrigado com 5 mL de água destilada, aspirado e seco com pontas de papel absorvente.

2.2. Condicionamento do substrato dentinário

Os espécimes utilizados foram distribuídos em 3 grupos (*n* = 20) experimentais, de acordo com o protocolo de condicionamento do substrato dentinário do espaço para pino. Em cada grupo, os espécimes foram distribuídos em 2 sub-grupos (*n*=10) de acordo com o período de análise (48 horas e 1 ano). Após o condicionamento ácido da dentina com ácido fosfórico a 37% (% (Condac 37; FGM, Joinville, SC, BR), por 15 segundos e irrigação com água destilada por 20 segundos, o conteúdo do espaço para pino foi aspirado e submetido a um dos seguintes protocolos:

AD (Água destilada): O espaço para pino foi preenchido com água destilada, mantida no local por 60 segundos e, após a aspiração do conteúdo com cânula de aspiração endodôntica (Cappilary tips;Ultradent, South Jordan, UT, USA), foi seco com pontas de papel absorvente;

AT (Ácido tânico): A solução aquosa de 5% tanino (Control-lab, Curitiba, PR, BR) foi utilizada similar ao descrito no DW protocol

GL (Glutaraldeído): A solução aquosa de 5% glutaraldehyde (Sigma-Aldrich, St Louis, MI, USA) foi utilizada similar ao descrito no DW protocol.

2.3. Cimentação do pino de fibra

Imediatamente após o condicionamento do substrato dentinário com um dos protocolos anteriormente descritos, o adesivo condiciona-e-lava (Adper Scocthbond Multipurpose; 3M ESPE, Saint Paul, MN, USA) foi aplicado na dentina do espaço para pino e fotoativado por 20 s. A superfície externa do pino de fibra (White Post DC # 1; FGM, Joinville, SC, BR) foi limpa com etanol a 95% (Rinse-N-Dry; Vista Apex, Racine WI, USA) e condicionada com ácido fosfórico a 37% (Condac 37; FGM, SC, BR) for 1 min. Em seguida, o silano (Prosil; FGM, Joinville, SC, BR) e o adesivo condiciona-e-lava (Adper Scocthbond Multipurpose; 3M ESPE, Saint Paul, MN, USA) foram sequencialmente aplicados e fotoativados por 60s (Valo; Ultradent, Salt Lake, UT, USA). O cimento resinoso convencional dual (RelyX ARC; 3M ESPE, Saint Paul, MN, USA) foi manipulado, conforme recomendações do fabricante, e inserido no espaço para pino.

Após o pino de fibra estar posicionado no espaço para pino com o sistema de cimentação, todo o conjunto foi submetido à fotoativação por 40s em cada face do espécime. Os espécimes foram armazenados em saliva artificial (FCFAr-Unesp, Araraquara, SP, BR). Dez espécimes de cada grupo foram submetidos ao teste de push-out após 48 horas e os outros 10 espécimes permaneceram imersos na saliva artificial, por 1 ano, a 37ºC, para somente então serem analisados.

2.4. Resistência de união

Os espécimes foram verticalmente posicionados e centralizados em uma matriz de PVC (21,3 mm de diâmetro × 20,0 mm de comprimento). A fim de manter a correta verticalização das raízes no interior das matrizes, as mesmas tiveram seus ápices radiculares fixados com cera em uma placa de vidro e o posicionamento conferido com um paralelômetro (BioArt B2, São Carlos, SP, BR). As matrizes foram preenchidas com resina poliéster (Maxi Rubber, Diadema,SP, BR), mantendo 1,0 mm do segmento cervical da raiz fora da inclusão. Todo o conjunto permaneceu intacto por 24 h, até que ocorresse a completa polimerização da resina. Após a desinclusão das matrizes de PVC, os espécimes foram seccionados perpendicularmente ao seu longo eixo, utilizando um disco diamantado adaptado em máquina de corte para tecidos duros (Isomet; Buehler Ltd, Lake Bluff, IL, USA), sob intensa refrigeração.

Três secções foram obtidas, com espessura de 2.0 mm + 0.1, dos terços apical, médio e cervical do espaço para pino de cada raiz. As secções cervical, média e apical foram obtidas respectivamente a partir de 3,0, 7,0 e 11,0 mm da face cervical da raiz.

As irregularidades das secções foram removidas com lixa d'água com granulação #1200 (Norton, São Paulo, SP, BR) e lavadas em câmara ultrassônica com água destilada, por 5 min.Os pinos de fibra de vidro cimentados no espaço protético de cada um dos terços das raízes foram submetidos ao teste de resistência de união (push-out) em máquina de ensaio eletromecânica (EMIC, São José dos Pinhais, PR, Brasil), calibrada na velocidade de 0,5 mm/min, até que ocorresse a completa ruptura do conjunto pino de fibra/sistema de cimentação da dentina do canal radicular. Os diâmetros do crosshead utilizados foram de 1,2, 0,9, e 0,5 mm, respectivamente para os terços cervical, médio e apical dos espécimes obtidos do espaço preparado para pino. Os valores da força (F) necessária para que ocorresse deslocamento do conjunto foi obtida em N (Newton) e, posteriormente transformada em resistência de união (MPa), por meio da fórmula: MPa = F/AD, onde AD corresponde à área de adesão, conforme descrito por Magro et al (2015).

2.5. Incidência de falha adesiva

O padrão de falha adesiva demonstrado foi analisado e classificado conforme descrito por Ramos et al. (2019): tipo 1 (adesiva 1), ocorre na interface entre pino de fibra e cimento resinoso; tipo 2 (adesiva 2), ocorre na interface entre sistema de cimentação e dentina; tipo 3 (coesiva), no sistema de cimentação; tipo 4 (mista), combinação de 2 ou mais tipos de falhas adesivas.

2.6. Analise estatística

Os dados obtidos nos períodos de avaliação de 48 horas e 1 ano de controle foram independentemente submetidos ao teste de Shapiro Wilk e aos de ANOVA one-way and Tukey tests, com nível de significância de 5% ($p = 0.05$). Os dados obtidos na análise do padrão de falha adesiva foram somente descritivos.

3. Resultados

3.1. Resistência de união

No período de controle de 48 horas, independentemente do terço do espaço para pino avaliado, GL e AT demonstraram maiores valores de resistência que DW ($p<0.05$), mas ambos foram similares entre si ($p>0.05$).

No período de 1 ano de controle, GL demonstrou o maior valor de resistência de união em relação a AT e AD ($p < 0.05$), que demonstraram valores similares entre si ($p > 0.05$), independentemente do terço do espaço para pino analisado.

A tabela 1 demonstra a média aritmética e desvio padrão dos valores de resistência de união (em megapascal) do sistema de cimentação com adesivo condiciona-e-lava e cimento resinoso dual convencional, em função dos terços protocolos de condicionamento do substrato dentinário e terço do espaço para pino, nos períodos de 48 horas e 1 ano de controle.

Tabela 1- Média aritmética e desvio padrão dos valores de resistência de união (em MPa) do sistema de cimentação com adesivo condiciona-e-lava e cimento resinoso dual convencional, em função dos terços protocolos de condicionamento do substrato dentinário e terço do espaço para pino, nos períodos de 48 horas e 1 ano de controle.

		DW	TA	GL
	cervical	11.20 ± 2.66^{b}	18.69 ± 1.76^{a}	19.36 ± 2.76^{a}
48 hs	middle	8.56 ± 1.51^{b}	16.21 ± 2.54^{a}	18.40 ± 4.94^{a}
	apical	8.40 ± 1.01^{b}	15.46 ± 2.11^{a}	16.21 ± 2.34^{a}
	cervical	7.40 ± 1.10^{b}	8.79 ± 1.05^{b}	15.86 ± 2.41^{a}
1 year	middle	7.01 ± 0.94^{b}	8.75 ± 0.71^{b}	14.70 ± 2.24^{a}
	apical	6.96 ± 0.65^{b}	8.25 ± 0.83^{b}	14.37 ± 1.29^{a}

^{ab} Letras diferentes na mesma linha indica diferença significante ($p < 0.05$). AD, protocolo com água destilada; AT, protocolo com tanino a 5%; GL, protocolo com glutaraldeído a 5%.

3.2. Padrão de falha adesiva

No período de controle de 48 horas, GL e AT demonstraram a maior incidência de padrão de falha adesiva tipo 4. Por outro lado, AD demonstrou a maior incidência do padrão de falha adesiva tipo 2.

No período de controle de 1 ano, GL demonstrou a maior incidência de falha tipo 3. TA demonstrou maior incidência de falha tipo 4 e AD demonstrou maior incidência de padrão de fratura tipo 2.

A tabela 2 demonstra a incidência da falha adesiva entre o sistema de cimentação de pino de fibra e a dentina do espaço para pino, em função do protocolo de condicionamento do substrato dentinário, nos períodos de 48 horas e 1 ano de controle.

Tabela 2- Incidência da falha adesiva entre o sistema de cimentação de pino de fibra e a dentina do espaço para pino, em função do protocolo de condicionamento do substrato dentinário, nos períodos de 48 horas e 1 ano de controle

		DW	TA	GL
	adesiva 1	0	0	0
48 hs	adesiva 2	18	3	1
	coesiva	5	10	14
	mista	7	17	15
	adesiva 1	0	0	0
1 year	adesiva 2	20	3	4
	coesiva	5	7	22
	mista	5	20	4

DW, protocolo com água destilada; TA, protocolo com tanino a 5%; GL, protocolo com glutaraldeído a 5%.

4. Discussão

As soluções de tanino e glutaraldeído, ambas na concentração de 5%, proporcionaram maiores valores de resistência de união do sistema de cimentação com o adesivo condiciona-e-lava e cimento resinoso dual convencional em comparação ao grupo controle (água destilada), no período de 48 horas de controle. Entretanto, após 1 ano de controle, somente a solução de glutaraldeído proporcionou o maior de valor de resistência de união, independentemente do terço do espaço para pino. Portanto, a hipótese H_0 foi rejeitada.

Os efeitos dos condicionadores do substrato dentinário foram avaliados pelo teste de push out, pois é um método consagrado para quantificar as consequências de um determinado procedimento sobre a interface de adesão (Ramos et al., 2019; Belizário et al., 2019; Lorenzetti et al,, 2019; Pane et al., 2013). Ainda que alguns fatores possam ter alguma interferência sobre os resultados, devido a força de deslocamento ser transversal ao sentido dos túbulos dentinários, simula as condições clínicas de falhas na cimentação dos pinos de fibra do canal radicular (Sudsangiam et al., 1999; Pane et al., 2013; Alencar et al., 2021).

A dimensão da amostra foi calculada fundamentada em um estudo piloto avaliando a resistência de união em que dois espécimes de cada grupo e terço foram utilizados. O valor do desvio padrão e erro amostral foram obtidos e a quantidade numérica da amostra para cada terço foi posteriormente obtida. Adicionalmente, a dimensão da amostra adotada está em concordância com estudos prévios que utilizaram similar metodologia (Ramos et al., 2018; Belizário et al, 2019; Lorenzetti et al., 2019).

O desvio de orientação da força aplicada durante a obtenção dos espécimes, principalmente devido ao desrespeito de alinhamento no momento da inclusão dos dentes na resina, ocasiona uma significativa variação nos resultados de resistência de união (Pane et al., 2013). Como o cálculo da área de adesão é dada pela fórmula: $A = \pi \cdot g \cdot (R + r)$, onde a trunk generatrix (g) é obtida a partir da fórmula $g = \sqrt{(R - r)^2 + (h)^2}$, caso o valor de h (circular straigth cone trunk height) não seja constante, por falta de uniformidade na obtenção dos espécimes, os valores de resistência de união serão severamente comprometidos (Guiotti et al., 2014; Magro et al., 2015). Com o objetivo de evitar esta variável, nós incluímos os espécimes em resina vinílica com auxílio de um paralelômetro, que foram rigorosamente seccionados transversalmente ao eixo axial radicular, na mesma espessura e individualmente conferidos com um paquímetro (precisão de 0.01mm).

O diâmetro do punch preferencialmente deve ocupar entre 50% a 83% do diâmetro do canal radicular (Nagas et al., 2011; Pane et al., 2013). A fim de evitar qualquer influência sobre os valores de resistência de união, nós utilizamos punch com diâmetros diferentes e compatíveis para cada segmento do espaço para pino, respectivamente 1.2mm, 0.9mm e 0.5mm para os terços cervical, médio e apical. Com isto, evitamos qualquer atrito com a dentina radicular e/ou a pontual deformação plástica do material em avaliação.

No período de controle de 48 horas, não houve diferença entre os valores de resistência de união proporcionados pelo condicionamento do substrato dentinário com a solução de tanino a 5% e glutaraldeído a 5%, mas estes foram superiores aos proporcionados pela irrigação com água destilada (controle). Entretanto, após 1 ano de controle, apenas a solução de glutaraldeído a 5% demonstrou maiores valores em comparação ao controle e até mesmo em relação à solução de tanino a 5%. Uma vez que o sistema de cimentação utilizado foi com o adesivo condiciona-e-lava, possivelmente dois fatores cruciais estiveram envolvidos e repercutiram podem ter influenciado nos resultados: a. *crosslink* das fibras colágenas e b. degradação progressiva das fibras colágenas (Belizário et al, 2018; Zheng et al., 2015)

As metaloproteinases (MMPs) e catepsinas são enzimas que participam do processo de maturação da dentina, no entanto, elas ficam presas e inativas após a matriz colágena se mineralizar (Malacarne et al., 2006). Durante as etapas do procedimento adesivo, as MMPs podem se tornar ativas (Pashley et al., 2004), uma vez que há desmineralização da parte mineral da dentina e elas serão ativadas (Sofan et al, 2008)). Os sistemas adesivos condicionam-e-lava contribuem para uma maior ativação dessas proteases pois possuem caráter ácido (Pashley et al., 2004). Uma vez que durante o processo de adesão, ser quase impossível envolver toda a rede de fibras colágenas com o adesivo, as mais profundas do colágeno podem ter ficado desprotegidas e envoltas em água, comprometendo a dentina submetida ao condicionamento com a solução de tanino a 5% , após o período de 1 ano de controle (Hashimoto et al., 2003; Carrilho et al., 2007; Zhou et l., 2009).

Por outro lado, os resultados similares entre as soluções de glutarladeído e tanino, ambas na concentração de 5%, no período de 48 horas, pode ser atribuída à imediata atividade de *crosslinker* proporcionada por estas substâncias (Belizário et al. 2019; Seseogullari-Dirihan et al. 2017). Porém, é possível que o *crosslink* proporcionado pela solução de tanino a 5% seja menos intensa e/ou passível de reversibilidade ao longo do tempo, em comparação à proporcionada pelo glutaraldeido, justificando os piores resultados após 1 ano de controle (Seseogullari-Dirihan et al., 2017).

Portanto, estudos futuros são necessários avaliar as concentrações, tempo de aplicação e composição química das soluções de glutaraldeído e de tanino, a fim de melhorar a longevidade da interface adesiva entre a dentina do espaço para pino e o sistema de cimentação com adesivo condiciona-e-lava e cimento resinoso convencional.

Conclusão

A solução de tanino a 5% não proporciona maior longevidade da interface adesiva entre a dentina do espaço para pino e o sistema de cimentação com adesivo condiciona-e-lava e cimento resinoso convencional.

Referências

1. Aranda-Garcia AJ, Kuga MC, Vitorino KR, Chávez-Andrade GM, Duarte MA, Bonetti-Filho I, Faria G, Só MV. 2013. Effect of the root canal final rinse protocols on the debris and smear layer removal and on the push-out strength of an epoxy-based sealer. Microscopy Research and Technique 76, 533-537.

2. Magro MG, Kuga MC, Aranda-Garcia AJ, Victorino KR, Chávez-Andrade GM, Faria G, Keine KC, Só MV. 2015. Effectiveness of several solutions to prevent the formation of precipitate due to the interaction between sodium hypochlorite and chlorhexidine and its effect on bond strength of an epoxy-based sealer. International Endodontic Journal 48, 478-483.

3. Sudsangiam S, van Noort R. 1999. Do dentin bond strength tests serve a useful purpose? The Journal of Adhesive Dentistry 1, 57-67.

4. Pashley DH, Tay FR, Yiu C, Hashimoto M, Breschi L, Carvalho RM, et al. 2004. Collagen degradation by host-derived enzymes during aging. J Dent Res 83, 216-21.

5. Lorenzetti CC, Bortolatto JF, Ramos ATPR, Shinohara AL, Saad JRC, Kuga MC. 2019. The effectiveness of glass ionomer cement as a fiber post cementation system in endodontically treated teeth. Microscopy Research and Technique 82:1191-1197.

6. Belizário LG, Kuga MC, Castro-Núñez GM, Escalante-Otárola WG, Só MVR, Pereira JR. Effects of different peracetic acid formulations on post space radicular dentin. J Prosthet Dent. 2018 Jan 5. pii: S0022-3913(17)30554-1.

7. Ramos ATPR, Garcia Belizário L, Venção AC, Fagundes Jordão-Basso KC, de Souza Rastelli AN, de Andrade MF, Kuga MC. 2018. Effects of photodynamic therapy on the adhesive interface of fiber posts cementation protocols. Journal of Endodontics 44, 173-178.

8. Belizário LG, Kuga MC, Hungaro Duarte MA, Só MVR, Keine KC, Pereira JR. 2019. Effect of fiber post space irrigation with different peracetic acid formulations on the bond strength and penetration into the dentinal tubules of self-etching resin cement. The Journal of Prosthetic Dentistry 122, 46.e1-46-7.

9. Alencar CM, Verbicário Dos Santos J, Jassé F, Dos Santos GO, Escalante-Otárola WG, Castro-Nuñez GM, de Campos EA, Kuga MC. 2021. Protocols for mechanical cleaning of the post space on the bond strength between root dentin and cementation system. Operative Dentistry 46, 467-475.

10. Malacarne J, Carvalho RM, de Goes MF, Svizero N, Pashley DH, Tay FR, et al. 2006. Watersorption/solubility of dental adhesiveresins. Dent Mater 22, 973-80.

11. Seseogullari-Dirihan R, Mutluay MM, Pashley DH, Tezvergil-Mutluay A. 2017. Is the inactivation of dentin proteases by crosslinkers reversible? Dental Material 33, e62-e68.

12. Carrilho MR, Carvalho RM, de Goes MF, di Hipólito V, Geraldeli S, Tay FR, et al. 2007. Chlorhexidine preserves dentin bond in vitro. Journal of Dental Research 2007 86, 90-4.

13. Polassi MR, Oliveira TS, de Carvalho AC, Medeiros LSM, Veiga TAM, Graeff CFO, González AHM, Marcucci MC, Grecco SDS, D'Alpino PHP. 2021. Influence of Dentin Priming with Tannin-Rich Plant Extracts on the Longevity of Bonded Composite Restorations. Scientific World Journal. 15;2021:1614643.

14. Hashimoto M, Ohno H, Sano H, Kaga M, Oguchi H. 2003. In vitro degradation of resin-dentin bonds analyzed by microtensile bond test, scanning and transmission electron microscopy. Biomaterials. 24, 3795-803.

15. Citta M, Anovazzi G, Basso FG, Scheffel D, Zhou J, Pashley DH, Souza Costa CA, Hebling J. 2021. Mechanical Stability and Proteolytic Activity of Resin-dentin Bonds Using the Cross-linked Dry Bonding Technique. Operative Dentistry 46,17.

16. Alonso JRL, Basso FG, Scheffel DLS, de-Souza-Costa CA, Hebling J. 2018. Effect of crosslinkers on bond strength stability of fiber posts to root canal dentin and in situ proteolytic activity. Journal of Prosthetic Dentistry 119,:494.e1-494.e9.

17. Lee J, Sabatini C.2017. Glutaraldehyde collagen cross-linking stabilizes resin-dentin interfaces and reduces bond degradation.European Journal of Oral Science 2017 125, 63-71.

18. Pane ES, Palamara JE, Messer HH. 2013. Critical evaluation of the push-out test for root canal filling materials. Journal of Endodontics 39, 669-673.

19. Guiotti FA, Kuga MC, Duarte MA, Sant'Anna AJ, Faria G. 2014. Effect of calcium hydroxide dressing on push-out bond strength of endodontic sealers to root canal dentin. Brazilian Oral Research 28.

20. Nagas E, Uyanik O, Durmaz V, Cehreli ZC. 2011. Effect of plunger diameter on the push-out bond values of different root filling materials. International Endodontic Journal 44, 950-955.

21. Zheng P, Zaruba M, Attin T, Wiegand A.2015. Effect of different matrix metalloproteinase inhibitors on microtensile bond strength of an etch-and-rinse and a self-etching adhesive to dentin. Oper Dent 40:80-6.

22. Pashley DH, Tay FR, Yiu C, Hashimoto M, Breschi L, Carvalho RM, et al. 2004. Collagen degradation by host-derived enzymes during aging. J Dent Res 83, 216-21.

23. Sofan E, Sofan A, Palaia G, Tenore G, Romeo U, Migliau G. 2017. Classification review of dental adhesive systems: from the IV generation to the universal type. Ann Stomatol Jul 3;8, 1-17.

24. Zhou J, Tan J, Chen L, Li D, Tan Y. The incorporation of chlorhexidine in a two-step self-etching adhesive preserves dentin bond in vitro. J Dent. 2009 Oct;37(10):807-12.
25. Sallach RE, Cui W, Balderrama F, Martinez AW, Wen J, Haller CA, Taylor JV, Wright ER, Long RC Jr, Chaikof EL. Long-term biostability of self-assembling protein polymers in the absence of covalent crosslinking. Biomaterials. 2010 Feb;31(4):779-91.

26. Comba A, Scotti N, Mazzoni A, Maravic T, Ribeiro Cunha S, Michelotto Tempesta R, Carossa M, Pashley DH, Tay FR, Breschi L. 2019. Carbodiimide inactivation of matrix metalloproteinases in radicular dentine. Journal of Dentistry 82:56-62.

3.2 PUBLICAÇÃO 2

Calcium hypochlorite to remove methylene blue after photodynamic therapy improves the bond strength to post space dentin*

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* Artigo redigido de acordo com as normas do Photodiagnosis and Photodynamic Therapy

Abstract

Introduction: 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.

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 like 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: Bond strength, fiber posts, glass-ionomer cement, photodynamic therapy,self-adhesive cement

Significance: PDT has emerged as a coadjuvant antimicrobial strategy in endodontic treatment.However, the effect of irrigants for photosensitizer removal on adhesive interface of fiber post and root dentin remains unclear and must be evaluated to establish effective protocols.

1. 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 ($Ca(OCl)_2$), which also has the ability to clarify tissues, has been purposed (11). Although $Ca(OCl)_2$ 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.

2. 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).

2.1. 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 4ºC, 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 37ºC 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.

2.2. 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/cm^2).

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.

2.3. Incidence of residues

2.3.1. 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 (AsferIndústriaQúimica, São Caetano do Sul, SP, Brazil) followed by aspiration (Cappilary 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.

2.3.2. 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 37ºC ± 1ºC 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.

2.4. Push-out bond strength

2.4.1. 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 that, 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/cm^2 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 37ºC ± 1ºC;

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).

2.4.2. 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 mm ± 1 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).

2.5. 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.

2.6. 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.

3. Results

3.1. Incidence of residues

Table 3 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 ($P < .05$), but similar to CH protocol ($P > .05$). On the other hand, SH protocol exhibited similar incidence of residues to CH protocol ($P > .05$). 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.

3.2. Push-out bond strength

Table 4 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 ($P < .05$), but they were similar to each other ($P > .05$). No difference between the other groups was observed ($P > .05$).

3.3. Failure mode incidence

Figure 1 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 2).

4. 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 3; 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.

5. 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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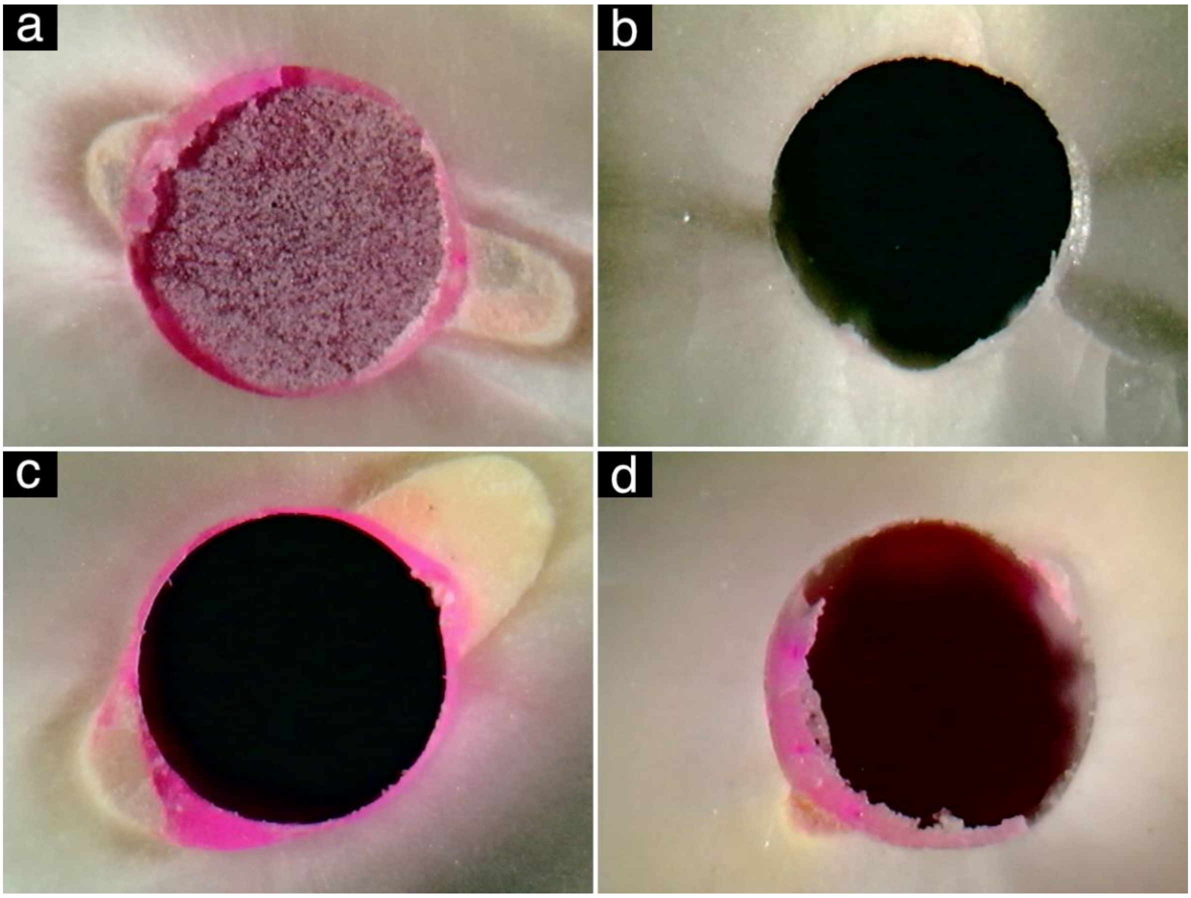
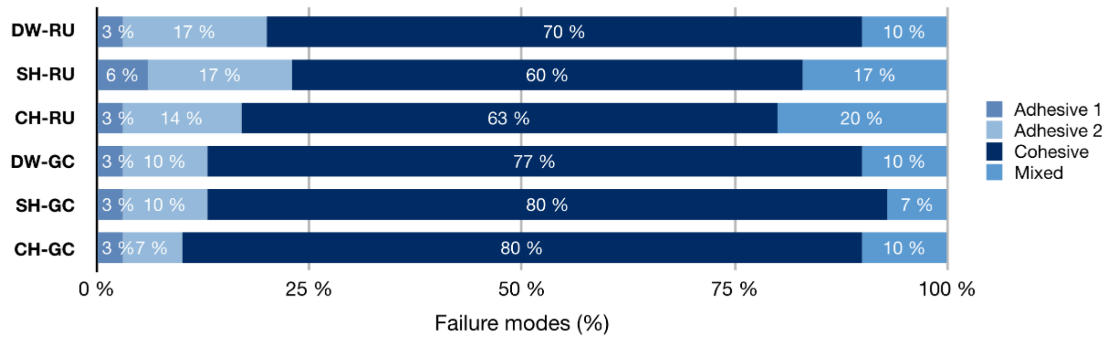
FIGURE LEGENDS

Figure 1- 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



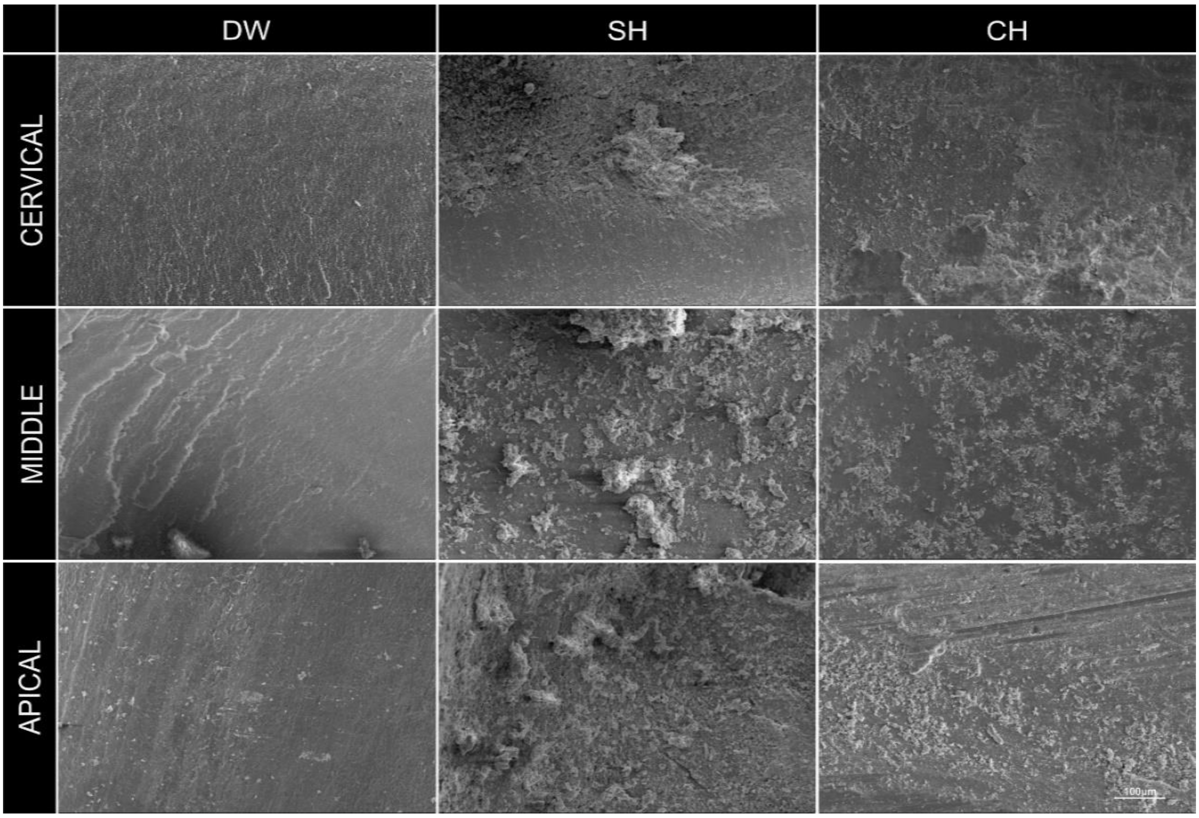
Fonte: Elaboração própria

Figure 2- 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.



Fonte: Elaboração própria

Figure 3- 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: 20µm.



Fonte: Elaboração própria

Table 3- Composition of the dental materials used in the cementation protocols.

Materials	**Manufacturer**	**Composition**
Relyx U200	3M ESPE, St Louis, MN, USA	Methacrylate
GC Gold Label 1 Luting & Lining	CG America, USA	Polybasic carboxylic acid
Silane	FGM, Joinville, SC, BR	MPS

Table 4- Median, maximum and minimum values of the attributed scores for the incidence of residuals on the dentin surface in function of the irrigation protocols to remove 0.01% methylene blue and the post space thirds.

		DW	SH	CH
cervical	**median** **max-min**	1^{a} (0-1)	2^{b} (1-2)	1^{ab} (1-2)
middle	**median** **max-min**	1^{a} (0-2)	2^{b} (2-2)	1^{ab} (1-2)
apical	**median** **max-min**	1^{a} (0-2)	2^{b} 2-2)	1.5^{ab} (1-2)

^{ab} Different letters within the same line 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 5- Mean and standard deviation of the push out bond strength values (MPa) in function of the 0.01% methylene blue removal protocol irrigation and the luting system, in 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.

References

1 Aihara HARAGUSHIKU Gisele, Donato Eing Engelke BACK Eduardo, Henrique TOMAZINHO Paulo, Baratto Filho Flares, Yoshio FURUSE Adilson. Influence of antimicrobial solutions in the decontamination and adhesion of glass-fiber posts to root canals. J Appl Oral Sci 2015;23(4):436–41. Doi: 10.1590/1678-775720150005.

2 Singh Shipra, Nagpal Rajni, Manuja Naveen, Tyagi Sashi Prabha. Photodynamic therapy: An adjunct to conventional root canal disinfection strategies. Aust Endod J 2015;41(2):54–71. Doi: 10.1111/aej.12088.

3 Trindade Alessandra Cesar, De Figueiredo José Antônio Poli, Steier Liviu, Weber João Batista Blessmann. Photodynamic Therapy in Endodontics: A Literature Review. Photomed Laser Surg 2015;33(3):175–82. Doi: 10.1089/pho.2014.3776.

4 Plotino G., Grande N. M., Mercade M. Photodynamic therapy in endodontics. Int Endod J 2019;52(6):760–74. Doi: 10.1111/iej.13057.

5 Costa Larissa Menezes, Matos Felipe De Souza, Correia Ayla Mac Yelle De Oliveira, et al. Tooth color change caused by photosensitizers after photodynamic therapy: An in vitro study. J Photochem Photobiol B Biol 2016;160:225–8. Doi: 10.1016/j.jphotobiol.2016.04.019.

6 Figueiredo Rômulo Aguiar, Anami Lilian Costa, Mello Isabel, Carvalho Erica Dos Santos, Habitante Sandra Márcia, Raldi Denise Pontes. Tooth discoloration induced by endodontic phenothiazine dyes in photodynamic therapy. Photomed Laser Surg 2014;32(8):458–62. Doi: 10.1089/pho.2014.3722.

7 Carvalho Erica Dos Santos, Mello Isabel, Albergaria Silvio José, Habitante Sandra Márcia, Lage-Marques José Luiz, Raldi Denise Pontes. Effect of chemical substances in removing methylene blue after photodynamic therapy in root canal treatment. Photomed Laser Surg 2011;29(8):559–63. Doi: 10.1089/pho.2010.2922.

8 Ramalho Karen Müller, Cunha Sandra Ribeiro, Mayer-Santos Eric, et al. In vitro evaluation of methylene blue removal from root canal after Photodynamic Therapy. Photodiagnosis Photodyn Ther 2017;20(October):248–52. Doi: 10.1016/j.pdpdt.2017.10.024.

9 Alencar Cristiane De Melo, Costa Joatan Lucas de Sousa Gomes, Jassé Fernanda Ferreira de Albuquerque, de Campos Edson Alves, Dantas Andréa Abi Rached, Kuga Milton Carlos. Evaluation of various methods of methylene blue removal from the post space after photodynamic therapy on the bonding interface using different resin cementation systems. Photodiagnosis Photodyn Ther 2021;34(February):1–8. Doi: 10.1016/j.pdpdt.2021.102264.

10 Ramos Anna Thereza Peroba Rezende, Garcia Belizário Lauriê, Venção Ana Carolina, et al. Effects of Photodynamic Therapy on the Adhesive Interface of Fiber Posts Cementation Protocols. J Endod 2018;44(1):173–8. Doi: 10.1016/j.joen.2017.08.035.

11 Görduysus Melahat, Küçükkaya Selen, Bayramgil Nursel Pekel, Görduysus Mehmet Ömer. Evaluation of the effects of two novel irrigants on intraradicular dentine erosion, debris and smear layer removal. Restor Dent Endod 2015;40(3):216. Doi: 10.5395/rde.2015.40.3.216.

12 De Almeida Ana Paula, Souza Matheus Albino, Miyagaki Daniela Cristina, Dal Bello Yuri, Cecchin Doglas, Farina Ana Paula. Comparative evaluation of calcium hypochlorite and sodium hypochlorite associated with passive ultrasonic irrigation on antimicrobial activity of a root canal system infected with Enterococcus faecalis: An in vitro study. J Endod 2014;40(12):1953–7. Doi: 10.1016/j.joen.2014.08.025.

13 Khoroushi Maryam, Amirkhani Najafabadi Maryam, Feiz Atiyeh. Effects of Calcium Hypochlorite and Sodium Hypochlorite, as Root Canal Irrigants, on the Bond Strength of Glass Fiber Posts Cemented with Self-Adhesive Resin Cement. Front Dent 2019;16(3):214–23. Doi: 10.18502/fid.v16i3.1593.

14 Dal Bello Yuri, Mezzalira Gean I., Jaguszewski Luis A., et al. Effectiveness of calcium and sodium hypochlorite in association with reciprocating instrumentation on decontamination of root canals infected with Enterococcus faecalis. Aust Endod J 2019;45(1):92–7. Doi: 10.1111/aej.12289.

15 Lorenzetti Camila C., Bortolatto Janaina F., Ramos Anna T.P.R., Shinohara André L., Saad Jose R.C., Kuga Milton C. The effectiveness of glass ionomer cement as a fiber post cementation system in endodontically treated teeth. Microsc Res Tech 2019;82(7):1191–7. Doi: 10.1002/jemt.23268.

16 Pereira Kamila de Figueiredo, Venção Ana Carolina, Magro Mirian G, et al.Effect of endodontic retreatment on the bond strength of resin cements to root canal dentin. Am J Dent 2019;32(3):147–51.

17 Aranda-garcia Arturo Javier, Kuga Milton Carlos, Vitorino Keli Regina, Antonio Marco, Duarte Hungaro. Effect of the Root Canal Final Rinse Protocols on the Debris and Smear Layer Removal and on the Push-Out Strength of an Epoxy-Based Sealer. Microsc Res Tech 2013;537(February):533–7. Doi: 10.1002/jemt.22196.

18 Serafino Cinzia, Gallina Giuseppe, Cumbo Enzo, Ferrari Marco. Surface debris of canal walls after post space preparation in endodontically treated teeth: A scanning electron microscopic study. Oral Surg Oral Med Oral Pathol Oral Radiol Endod 2004;97(3):381–7. Doi: 10.1016/j.tripleo.2003.10.004.

19 Ramos Anna Thereza Peroba Rezende, Belizário Lauriê Garcia, Jordão-Basso Keren Cristina Fagundes, Shinohara André Luis, Kuga Milton Carlos. Effects of photodynamic therapy on the adhesive interface using two fiber posts cementation systems. Photodiagnosis Photodyn Ther 2018;24(April):136–41. Doi: 10.1016/j.pdpdt.2018.08.017.

20 Magro M G, Kuga M C, Victorino K R, Faria G, Keine K C. Effectiveness of several solutions to prevent the formation of precipitate due to the interaction between sodium hypochlorite and chlorhexidine and its effect on bond strength of an epoxy-based sealer. Int Endod J 2014:1–6. Doi: 10.1111/iej.12337.

21 Peroba Rezende Ramos Anna Thereza, Fagundes Jordão-Basso Keren Cristina, Porto Thiago Soares, Kuga Milton Carlos. Effect of irrigation protocol during post space preparation on the dentin adhesive interface: An in vitro study. J Prosthet Dent 2021;125(2):324.e1-324.e9. Doi: 10.1016/j.prosdent.2020.09.024.

22 Hülsmann Michael, Rümmelin Claudia, Schäfers Frank. Root canal cleanliness after preparation with different endodontic handpieces and hand instruments: A comparative SEM investigation. J Endod 1997;23(5):301–6. Doi: 10.1016/S0099-2399(97)80410-4.

23 Oliveira Lilian Vieira, Maia Thais Souza, Zancopé Karla, de Souza Menezes Murilo, Soares Carlos José, Moura Camilla Christian Gomes. Can intra-radicular cleaning protocols increase the retention of fiberglass posts? A systematic review. Braz Oral Res 2018;32. Doi: 10.1590/1807-3107bor-2018.vol32.0016.

24 Belizário Lauriê Garcia, Kuga Milton Carlos, Hungaro Duarte Marco Antônio, Só Marcus Vinicius Reis, Keine Kátia Cristina, Pereira Jefferson Ricardo. Effect of fiber post space irrigation with different peracetic acid formulations on the bond strength and penetration into the dentinal tubules of self-etching resin cement. J Prosthet Dent 2019;122(1):46.e1-46.e7. Doi: 10.1016/j.prosdent.2019.03.020.

25 Leonardo Natália Gomes E.Silva, Carlotto Israel Bangel, Luisi Simone Bonato, Kopper Patrícia Maria Poli, Grecca Fabiana Soares, Montagner Francisco. Calcium hypochlorite solutions: Evaluation of surface tension and effect of different storage conditions and time periods over pH and available chlorine content. J Endod 2016;42(4):641–5. Doi: 10.1016/j.joen.2016.01.006.

26 Belizário Lauriê Garcia, Kuga Milton Carlos, Castro-Núñez Gabriela Mariana, Escalante-Otárola Wilfredo Gustavo, Só Marcus Vinicius Reis, Pereira Jefferson Ricardo. Effects of different peracetic acid formulations on post space radicular dentin. J Prosthet Dent 2018;120(1):92–8. Doi: 10.1016/j.prosdent.2017.08.002.

27 Silva A., Alencar CM., Jassé FA., et al. Effect of post-space irrigation with acid solutions on bond strength and dentin penetrability using a self-adhesive cementation system. J Clin Exp Dent 2020;13(6):0–0. Doi: 10.4317/jced.58029.

28 Pane Epita S., Palamara Joseph E.A., Messer Harold H. Critical evaluation of the push-out test for root canal filling materials. J Endod 2013;39(5):669–73. Doi: 10.1016/j.joen.2012.12.032.

29 Blattes Gabriela Bess Ferraz, Mestieri Leticia Boldrin, Böttcher Daiana Elisabeth, Fossati Anna Cristina Medeiros, Montagner Francisco, Grecca Fabiana Soares. Cell migration, viability and tissue reaction of calcium hypochlorite based-solutions irrigants: An in vitro and in vivo study. Arch Oral Biol 2017;73:34–9. Doi: 10.1016/j.archoralbio.2016.08.037.

30 Seballos Vivian Gehm, Barreto Mirela Sangoi, da Rosa Ricardo Abreu, Machado Eduardo, Valandro Luiz Felipe, Kaizer Osvaldo Bazzan. Effect of post-space irrigation with NaOCL and CaOCL at different concentrations on the bond strength of posts cemented with a self-adhesive resin cement. Braz Dent J 2018;29(5):446–51. Doi: 10.1590/0103-6440201801955.

3.3 PUBLICAÇÃO 3

Effect of post space irrigation with 1% peracetic acid on bond strength and dentin penetrability using a self-adhesive cementation system*

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* Artigo redigido de acordo com as normas do Journal of Clinical and Experimental Dentistry.

Purpose: To evaluate the effects of surface treatments on post space root dentin with 1% peracetic acid (PA), Smear Clear or QMix solution compared to 2.5% sodium hypochlorite (NaOCl) on bond strength and dentin penetrability using a self-adhesive cementation system.

Materials and Methods: 40 human canines roots were endodontically treated and the post space was prepared. The specimens were randomized into four groups according to the surface treatment protocol (n = 10): Control – irrigation with 2.5% NaOCl solution; PA – irrigation with 1% peracetic acid; Smear Clear – irrigation with Smear Clear solution; and QMix – irrigation with QMix solution. Fiber posts were cemented with a self-adhesive resin system (Relyx U200). After six months, the specimens were cross-sectioned (1 slice for each third - cervical, middle, and apical) and submitted to push-out and confocal laser microscopy (CLSM) tests, respectively. The fracture pattern was analyzed with a stereomicroscope. For data analysis, one-way ANOVA and Tukey tests were applied ($\alpha = 0.05$).

Results: PA and QMix presented the highest values of bond strength when compared to the other groups ($p < 0.05$), regardless of the third analyzed. There was no significant difference between groups in the penetration of cement in the post space dentin ($p = 0.75$). Control group showed the highest incidence of adhesive type failure.

Conclusion: Treatment with 1% PA and QMix provided greater bond strength when compared to other irrigation protocols. However, they had no impact on the penetrability of the self-adhesive resin cement in the dentin of the post space.

Keywords: fiberglass post, adhesive interface, self-adhesive, root dentin.

Endodontically treated teeth show loss of coronal structure and decreased mechanical strength to intraoral chewing forces.[1] In a recent meta-analysis, fiber posts showed higher overall survival rates than metal posts when used to restore endodontically treated teeth with extensive coronal destruction.[2] Moreover, several characteristics are related with the high index of clinical choice for fiberglass posts such as more conservative removal of dentin, aesthetic advantage and greater agility in the treatment, since the cementation of this type of post does not require a laboratory step.[3,4]

Self-adhesive resin cements, such as RelyX U200 (3M ESPE, Saint Paul, MN, USA) were introduced in the market to reduce the sensitivity of the conventional three-step adhesive technique and avoid errors during the clinical protocol. Self-adhesive resin cements have presented equally effective when compared to conventional resin cement.[5] However, self-etching resin cement requires careful irrigation of the post space, with solutions that do not interfere with the bond strength and increase its penetrability in the dentinal tubules. A satisfactory adhesion of the posts to the root dentin is achieved when the smear layer is safely removed, as these residues can impair the adhesion.[6]

Therefore, chelating solutions play an important role in removing debris and the smear layer.[7]QMix (Dentsply Sirona Endodontics, Tulsa, OK, USA) is a commercial product indicated for irrigation and intra-canal cleaning. It aims to remove the inorganic smear layer and disinfect the root canal system. The chemical composition of QMix associates chelating properties of ethylenediaminetetraacetic acid (EDTA) with the substantive and antimicrobial properties of chlorhexidine gluconate.[8] In addition, it is composed of a mixture of antimicrobial agent from bisbiguanide, calcium from polyamine carboxylic acid, saline and a surfactant.[9] It was previously demonstrated that QMix as a cleaning solution showed less decalcification and erosion than 17% EDTA.[10] However, further studies and comparisons must be carried out to fully clarify this issue.

SmearClear (Sybron Endo, Orange, CA, USA) is a commercial product that was introduced in the market to assist in removing the smear layer. It is a 17% EDTA solution combined with a cationic (cetrimide) and anionic surfactant, with the aim of increasing its bacterial effectiveness and improving its clinical performance.[7] However, the evidence is controversial about the positive impact of this irrigating solution on dentin adhesion.[7,11]

Peracetic acid (PA) has shown to be a promising alternative to dissolve the smear layer and disinfect the root canal system.[12] This solution is sporicidal, bactericidal, virucidal and fungicidal in low concentrations. It breaks down into safe by-products and oxygen, reducing the smear layer formation as effectively as EDTA.[13] A recent previous study showed that 1% PA without association with other solutions provided a similar reduction in microhardness and roughness of root canal dentin, compared to EDTA and sodium hypochlorite (NaOCl). However, 1% PA caused less dentin erosion when compared to these irrigating solutions.[12] The number of studies available in the literature about the use of 1% PA as a single endodontic irrigating solution in the preparation of the post space is still small. Furthermore, the variety of irrigating endodontic solutions requires as many comparisons as possible to provide consistent evidence.

Therefore, the objectives of this *ex vivo* study were to evaluate the effect of surface treatment with 1% PA compared to other irrigating solutions on the bond strength between root dentin and fiber posts cemented with self-adhesive resin cement, and also to verify the penetrability of cement in the intratubular dentin after post-space treatment. The null hypotheses of this study were: H01 – There is no influence of the endodontic irrigation solutions used for post-space preparation on the bond strength between root dentin and fiber posts cemented with self-adhesive resin; and H02 – There is no influence of the endodontic irrigation solutions used in post-space preparation on the penetrability of the self-adhesive cement in the intratubular root dentin.

MATERIALS AND METHODS

Sample preparation

This study was approved by the Human Research Ethics Committee of the Araraquara Dental School (registration number: 44018715.9.0000.5416). Forty canines with similar root anatomy and extracted for periodontal reasons were extracted. Periapical radiographs were acquired in the mesiodistal and buccolingual directions to select teeth with a single canal. Teeth that presented immature apexes, root caries, root fractures, cracks, lacerations, sharp curvatures, canal calcifications or endodontic treatment were excluded; these features were identified with the aid of a stereomicroscope (SMX800, Nikon Co., NY, USA) under 20x magnification. Then, the specimens were stored in a 0.1% thymol solution at 4 ºC until use. After rinsing with distilled water, teeth were cross sectioned by using a diamond disk under water-cooling (Isomet 2000; Buehler Ltd., Lake Buff, IL, USA). All roots were standardized to a length of 17 mm.

A single operator performed the instrumentation of the root canals. Canal patency was performed with a 10 size K-file (Dentsply Maillefer, Ballaigues, Jura-Nord Vaudois, Switzerland) and the Path-File instruments 1, 2, 3 (Dentsply Maillefer, Ballaigues, Jura-Nord Vaudois, Switzerland). The working length (WL) of the canals was standardized at 16 mm. The root canals were prepared using the Universal ProTaper System (Dentsply Maillefer, Ballaigues, Jura-Nord Vaudois, Switzerland). First, the cervical and middle thirds of the roots were prepared using the instruments S1, SX and S2. After, S1, S2, F1, F2, F3, F4 and F5 files were sequentially used in WL. Finally, the canals were irrigated with 5 ml of 2.5% NaOCl (Asfer, São Caetano do Sul, SP, Brazil). The foraminal openings were sealed with composite resin to prevent extrusion of NaOCl during the irrigation procedures. After canal preparation, the specimens were irrigated with 5 mL of 17% EDTA (Biodinâmica, Ibiporã, PR, Brazil) for 3 minutes, and were subsequently rinsed with 5 mL of 2.5% NaOCl and completed with 5 mL of saline solution using Navitip needles (Ultradent Products Inc., South Jordan, UT) taken up to 3 mm short of the WL.

All the root canals were then dried by using F5 paper points (Dentsply Maillefer, Ballaigues, Jura-Nord Vaudois, Switzerland) and filled by the single cone technique (F5 gutta-percha point, Dentsply Maillefer) and an epoxy resin-based sealer (AH Plus; Dentsply De Trey, Konstanz, Baden-Württemberg, Germany). The endodontic sealer was mixed according to the manufacturer’s instructions and placed at the WL by means of a 400-rpm Lentulo spiral (Dentsply Maillefer, Ballaigues, Jura-Nord Vaudois, Switzerland) for 5 seconds. The excess gutta-percha in the coronal portion was removed with a flame-heated plugger, and the access cavity was sealed (Coltosol, Coltene, Rio de Janeiro, RJ, Brazil). All roots were stored immersed in artificial saliva (Faculdade de CiênciasFarmacêuticas-UNESP, Araraquara, SP, Brazil) at 37 ^{o}C for 7 days. Afterwards, they were included in plastic matrices as previously described by Victorino and others.[14]

After 24h, the root canal filling was partially removed using the Largo 1 to 4 drill sequence (Dentsply Maillefer, Ballaigues, Jura-Nord Vaudois, Switzerland), alternately with saline solution irrigation, in the length of 11 mm. Post space preparation was performed using the White Post #2 bur (White Post DC, FGM, Joinville, SC, Brazil) at 11 mm. Periapical radiographs were performed to verify that root filling was correctly removed.

Treatment with irrigation solutions

Samples were randomized into 4 groups (n = 10) according to the irrigation solution used during post-space preparation: 2.5% NaOCl (control group); 1% PA (Peresal, Profilática, Araucária, PR, Brazil); Smear Clear (Kerr, Orange, CA, USA);

and QMix (Dentsply Tulsa Dental, Tulsa, OK, USA). The post-space was irrigated and aspired with 5 mL of the tested solution for 1 minute and the solution remained inside the root canal for 3 minutes in Group 2.5% NaOCl and Group 1% PA; for 1 minute in Group Smear Clear, and for 2 minutes in Group QMix, according to the manufacturer's recommendations. After, the post-spaces were rinsed with 5 mL of saline solution and dried with capillary tips, 0.48 mm in diameter (Capillary Tips; Ultradent, South Jordan, UT, USA) and F5 paper points.

Glass fiber posts #2 (White Post DC, FGM, Joinville, SC, Brazil) were cleaned with 70% ethyl alcohol, coated with silane (Prosil, FGM, Joinville, SC, Brasil) and put out to dry for 1 minute, to allow solvent evaporation.

Fiberglass post cementation

The Rhodamine B dye (Sigma-Aldrich, St. Louis, MO, USA), in the proportion of 0.1% (m/m), was mixed with the self-adhesive resin cement (RelyX U200, 3M ESPE, St. Paul, USA), to provide the fluorescence that would enable confocal laser microscopy (CLSM) assessment. The cement was handled according to the manufacturer’s specification and inserted into the post space using Automix tips (3M ESPE, St. Paul, USA). Then, the post was positioned in the post space by manual pressure. The excess resinous cement was removed with a microbrush and cured for 40 seconds using an LED light curing unit (LED Bluephase, IvoclarVivadent, Schan, AL, Liechtenstein) with intensity of 1.200 mW/cm^2. The light guide tip of the light-curing unit was placed perpendicular to the post. Specimens were stored in distilled water at 37 ^{o}Cfor 6 months.

Bond strength and failure modes evaluation

After the immersion period, all roots were cross sectioned using a diamond disk under water-cooling in a cutting machine (Isomet, Buehler Ltd, Lake Bluff, IL, USA). Three slices with thickness of 2.0 mm $\pm$ 0.1 mm were obtained from each specimen. Cervical, middle and apical slices were obtained at 1, 5 and 8 mm, respectively, from the cervical root surface. Irregularities generated during sectioning procedures were removed with 1.200 water-wet sandpapers (3M ESPE, Saint Paul, MN, USA) in a polishing machine (Buehler, Lake Bluff, Illinois, USA).

The universal testing machine (EMIC, São José dos Pinhais, PR, Brazil) was used to push-out test at a crosshead speed of 0.5 mm/min. The slices were inserted into a metal device with a central hole (Ø = 3 mm) larger than the diameter of the canal. The coronal face of the specimen was placed face down and a metallic cylinder (Ø extremity = 0.8 mm) induced a load onto the pole from apical to coronal direction. The bond strength values (σ) were obtained in MPa: $\sigma = F/A$, where F = load for breaking the sample (n) and A = bonded area (mm^2). To determine the bonded interface area, a formula was used: $A = 2\pi g (R_1 + R_2)$, where $\pi = 3.14$, g = slant height, R_1 = smaller base radius, R_2 = larger base radius. To determine the slant height, the following calculation was used: $g_2 = [h_2 + (R_2 - R_1)_2]$, where h= section height. The measures of R_1 and R_2 were obtained through the internal diameters of the minor and major bases, respectively. The diameters and h were measured using a digital caliper (Starrett 727, Starrett, Itu, SP, Brazil).

Dentine slices were analyzed under stereomicroscopy (Zeiss Stemi SV6) at 20× magnification in order to categorize the failure modes, according to Elnaghy,[15] as follows: Type 1 – Adhesive failure at fibre post/cement interface; Type 2 – Cohesive failure at inside the cement; Type 3 – Adhesive failure at dentin/cement interface; and Type 4 – Mixed failure occur in combination.

Intratubular cement penetration

After the push-out test, intratubular cement penetration was examined with the Olympus FluoView Confocal Laser 1000 Microscope (Olympus Corporation, Tokyo, Japan). Cervical, middle, and apical samples were assessed. The absorption and emission wavelengths for Rhodamine B were 540 nm and 494 nm, respectively. Dentine samples were analyzed using the 10× oil lens at 100× magnification. The image settings were 70-µm depth with 800 × 800 pixels. The Adobe Photoshop software (Adobe Systems, San Jose, CA, USA) was use for image analysis as previously described.[16] Initially, the overall area of the image was obtained. Next, the lasso tool was used to outline and measure the canal area. By subtracting these values, the dentine area was obtained. Thus, the sealer impregnated dentine area was outlined with the same tool and the percentage of sealer penetration was calculated.

Statistical analysis

SPSS statistical software was used as analytical tool (SPSS 12.0; SPSS Inc., Chicago, IL, USA). The normality of the data was verified using the Shapiro Wilk test that confirmed normal distribution of the data W = 0.842 and W = 0.832 for bond strength and intratubular cement penetration, respectively. Thus, these outcomes were analyzed by using one-way ANOVA and Tukey tests ($\alpha = 0.05$).

RESULTS

Bond strength and failure modes evaluation

For bond strength evaluation, one-way ANOVA indicated significant differences between the experimental groups in all post space thirds ($p < 0.05$). 1% PAand QMix presented the highest bond strength mean values when all thirds were grouped and evaluated ($p < 0.05$). In the cervical and middle thirds, 1% PA and QMix promoted higher bond strength values than 2.5% NaOCl and Smear Clear (p <

0.05). In the apical third of the post space, QMix presented the highest bond strength values, followed by 1% PA ($p < 0.05$). The lowest bond strength was found in Group 2.5% NaOCl ($p < 0.05$). Tables 6-8 shows the mean and standard deviation of bond strength values for all groups in each post space third.

Adhesive failures between dentine/cement (N = 34) and adhesive failures between post/cement (N = 33) were predominant, followed by mixed failures (N = 28) and cohesive failure of the cement (N = 25). Additionally, numerous adhesive failures occurred at the dentine/cement interface in Group 2.5% NaOCl.

Intratubular cement penetration

After assessment of intratubular cement penetration, one-way ANOVA found no differences among the groups, irrespective of the post space third evaluated ($p = 0.85$) (Figure 4).

DISCUSSION

Considering the need for further studies related to this topic, the present study investigated the effect of different intracanal irrigants on bond strength between root dentine and fiber posts cemented with self-adhesive resin cement, and the intratubular cement penetration after post space irrigation with different solutions. In view of the results, the first null hypothesis was rejected. The irrigant used for post space irrigation influenced the bond strength to root dentine of fiber posts cemented with self-adhesive resin cement. On the other hand, the second null hypothesis was confirmed. The irrigant used for post space irrigation did not affect the intratubular cement penetration.

The manufacturer of RelyX U200 recommends irrigating the post-space preparation with NaOCl prior to post-cementation of the fiber; however, the criteria used to choose this irrigant are not clear. Two recent studies have shown that post space irrigation with 2.5% NaOCl has not shown the best bond strength results when compared to other alternative treatments.[6,17] However, Barreto et al[7] found higher bond strength values when saline and 2.5% NaOCl were used and recommended these solutions for post space cleaning before post cementation when using self-adhesive cements. It must be highlighted that in the cited study, the authors performed passive ultrasonic irrigation (PUI). In the present study, PUI was not performed, furthermore, the irrigation solutions were used according to the manufacturer's recommendations thus the real effect of the solutions on root dentine was assessed.

The literature reported that the effect of NaOCl on organic dentin compounds has the potential to promote collagen degradation, negatively affecting the resistance of the fiber posts to root dentin.[18] In addition, Zhang et al[19] stated that when dentin was exposed to high concentrations of NaOCl for long periods, its bonding and flexural strength were impaired. Therefore, the time of dentin exposure to NaOCl and the concentration of NaOCl showed a negative correlation when compared to the values of adhesive strength.[15,19,20] In the present study, an intermediate concentration of NaOCl (2.5%) was used and the exposure time was three minutes. Thus, this time was able to promote post-space cleaning and some collagen degradation since the values of bond strength were low in all evaluated post-space thirds.

The QMix solution and PA showed the highest bond strength in the cervical and middle thirds of the post space; and in the apical third, only QMix showed the best adhesive bonding results. This can be explained due to the chemical composition and mechanism of action of this material on dentin. QMix contains EDTA, chlorhexidine and a detergent in its formulation which guarantees its low surface tension and increases its wettability and consequently, the flow of the

irrigation solution in the root canal in contact with the smear layer and the underlying dentin.[21] Probably, this activity occurs due to the mixture of chlorhexidine and detergent[22], resulting in the highest bond strength values with this solution use.

The literature has reported some studies that aimed to evaluate the effect of active irrigation using ultrasound and neodymium: yttrium-aluminum-garnet (Nd: YAG) laser to remove smear layer and debris after post space preparation.[7,23,24] Some studies have shown better cleaning of the root canal walls when ultrasonic and Nd: YAG activation was performed, mainly in association with chelating solutions.[25,26] On the other hand, some authors did not observe an improvement in the removal of smear layer and debris when active irrigation was performed.[23,27] The present study aimed to evaluate only the chemical effect promoted by the chelating solutions and did not include another variable such as PUI or Nd: YAG laser.

Among the various descaling agents available, peracetic acid appears to be a potential alternative among other chelating solutions. PA contains and releases acetic acid, which is a weak chelating agent, but capable of reducing the smear layer with the same effectiveness as EDTA, for example.[12,13] According to the authors' best knowledge to date, only one study is available in the literature on the use of peracetic acid before post-cementation and its impact on the bond strength of cemented fiber posts with self-adhesive cements.[6] However, 1% PA had greater cytotoxic potential than 2.5% NaOCl and is a disadvantage of this solution[28]. Considering this, further investigations and comparisons with different solutions are encouraged to increase the evidence on this issue. In relation to Smear Clear, a previous study showed that it do not result in better smear layer removal compared with EDTA alone,[29] which can be linked with the present founds since Smear Clear and 2.5% NaOCl presented statistical similar bond strength values. The contact times of the solutions with the dentin was performed according to the manufacturer's recommendations, so that no erosive data would occur on the dentin surface.

The failure patterns were variable in all groups. An interesting finding was that Group NaOCl presented adhesive failures at the cement/dentine interface in fifty percent of the specimens. This result may indicate an inadequate level of chemical bonding between the self-adhesive cement and root dentine caused by the degradation of collagen promoted by NaOCl.[18] One may argue that Group NaOCl should have been submitted to a final irrigation with 17% EDTA. However, the aim of this protocol was to create a negative control with a remain smear layer, since studies have been reported that when EDTA is used as a solvent for inorganic matter after preparation of the root canal with NaOCl, it dissolves the sparse “ghost mineral layer" in collagen and also exposes the underlying dentin, which is irreversibly destroyed by NaOCl.[30] So, we can hypothesize that an alternated irrigation protocol with NaOCl and EDTA causes more deleterious effects on dentinal tissues than unique solution protocols with disinfection and chelating activities, which could jeopardize our results. This is the reason for choosing of 2.5% NaOCl solution.

According to the findings of Kuga et al,[31] no differences were observed in the association of NaOCl with acid solutions over the depth of penetration into the root dentin. CLSM is often used in endodontics to determine the degree of adaptation and penetration of the root canal filling into dentin walls and dentinal tubules, respectively.[32] According to the CLSM images of the present study, no correlation was observed between cement penetration and bond strength values, including the 2.5% NaOCl group, which can be related to the time of exposure and renovation dependent character of this solution, such as above mentioned. However, Barreto et al[7] showed that EDTA and NaOCl associated with ultrasound activation proved to be promising solutions for cleaning root canals before cementing a fiberglass post with self-adhesive resin cement. Additional studies must be carried out to establish the relationship between the continuity in depth of the penetration of the self-adhesive resin cement and the results of bond strength. In addition, further *in vivo* studies are needed to assess the long-term effect of different post-space treatments on bond strength of adhesive materials to root dentin.

CONCLUSION

Peracetic acid at 1% and QMix showed the highest values of bond strength, while 2.5% NaOCl showed the lowest values. The failure patterns were varied, with high adhesive failures at the dentin/cement interface for NaOCl and Smear Clear root surface treatment. The penetration of cement into the dentinal tubules was not affected by the solution used to rinse the post space.

Table 6 - Mean and standard deviation of the bond strength values (MPa) for all groups in each post-space third.

Groups	Cervical	Middle	Apical	Overall
2.5% NaOCl	$1.77^{b} \pm 0.86$	$2.46^{b} \pm 1.22$	$1.61^{c} \pm 0.35$	$1.95^{b} \pm 0.93$
1% Peracetic Acid	$3.85^{a} \pm 1.4$	$4.15^{a} \pm 1.59$	$3.26^{b} \pm 0.55$	$3.75^{a} \pm 1.28$
Smear Clear	$1.94^{b} \pm 0.62$	$2.14^{b} \pm 1.1$	$2.81^{b} \pm 0.43$	$2.29^{b} \pm 0.83$
QMix	$4.44^{a} \pm 1.44$	$4.61^{a} \pm 1.45$	$4.22^{a} \pm 0.67$	$4.42^{a} \pm 1.21$

Different superscript letters indicate statistically significant differences between groups and thirds (5% significance level; one-way ANOVA and Tukey tests).

Table 7 – Failure mode presented in each experimental group after push-out test.

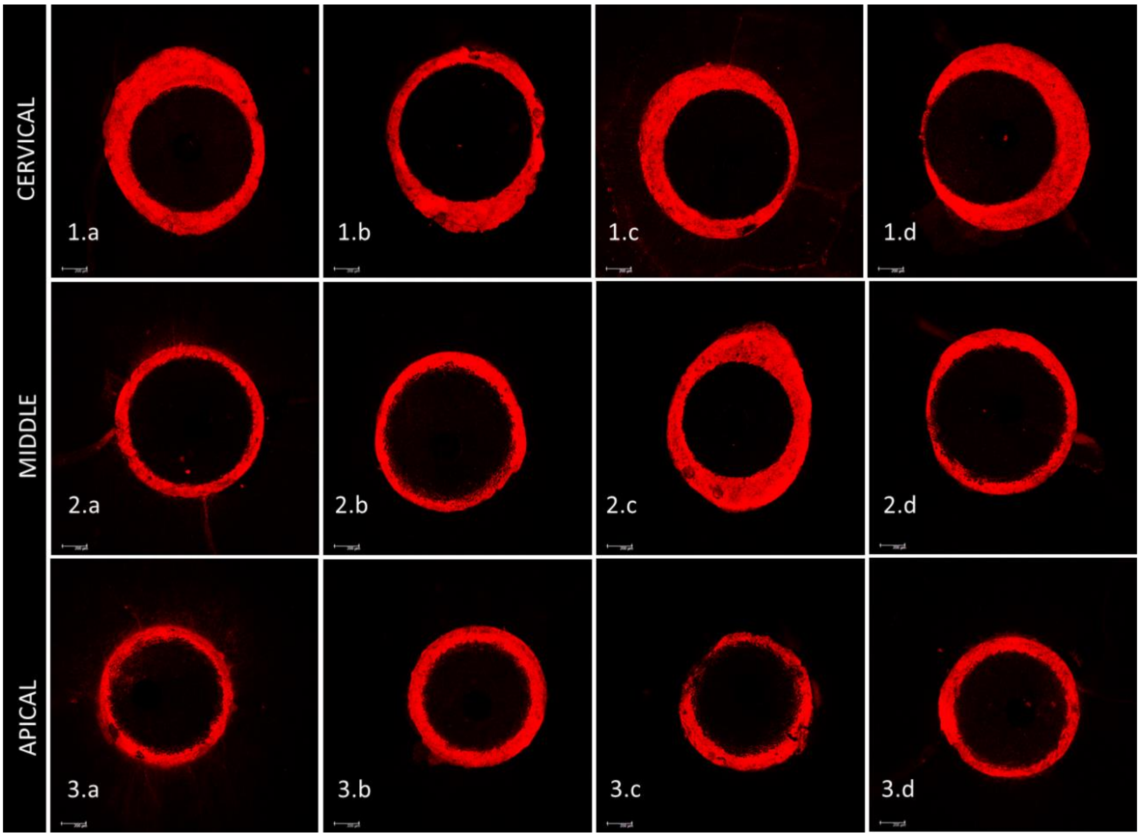
	2.5% NaOCl	Smear Clear	1% Peracetic Acid	QMix	Overall
Adhesive Post/Cement	5	6	10	12	33
Cohesive of the cement	3	6	6	10	25
Adhesive Dentin/Cement	15	8	6	5	34
Mixed	7	10	8	3	28
	30	30	30	30	120

Table 8 – Mean and standard deviation (%) of intratubular resin cement (U200) penetration after final irrigation of post space.

Groups	Cervical	Middle	Apical	Overall
2.5% NaOCl	$8.12^{a} \pm 4.03$	$10.51^{a} \pm 4.41$	$10.12^{a} \pm 3.53$	$9.61^{a} \pm 3.58$
1% Peracetic Acid	$6.37^{a} \pm 3.39$	$8.73^{a} \pm 3.46$	$12.46^{a} \pm 4.96$	$9.18^{a} \pm 3.87$
Smear Clear	$9.01^{a} \pm 1.80$	$10.17^{a} \pm 6.25$	$11.58^{a} \pm 3.40$	$10.54^{a} \pm 2.98$
QMix	$8.77^{a} \pm 3.20$	$12.94^{a} \pm 5.93$	$9.76^{a} \pm 2.69$	$10.49^{a} \pm 4.02$

Different superscript letters indicate statistically significant differences between groups and thirds (5% significance level: one-way ANOVA and Tukey tests)

Figure 4- Photomicrographs of the cervical, middle and apical thirds according to the surface treatment: 1. Cervical third: (1.a) - 2.5% NaOCl; (1.b) - 1% Peracetic acid; (1.c) - 17% EDTA; (1.d) - QMix solution; 2. Middle third: (2.a) 2.5% NaOCl; (2.b) 1% Peracetic acid; (2.c) - 17% EDTA; (2.d) - QMix solution; 3. Apical third: (3.a) 2.5% NaOCl; (3.b) 1% peracetic acid; (3.c) - 17% EDTA; (3.d) - QMix solution.



Fonte: Elaboração própria

References

1. Almohareb T.Sealing Ability of Esthetic Postand Core Systems. J Contemp Dent Pract.2017;18:627-632.

2. Pedro FM, Marques A, Pereira TM, Bandeca MC, Lima S, Kuga MC,Tonetto MR, Semenoff-Segundo A, Borges AH. Status of Endodontic Treatment and the correlations to the Quality of Root Canal Filling and Coronal Restoration.J Contemp Dent Pract.2016;17:830-836.

3. Ramos ATPR, Garcia Belizário L, Venção AC, Fagundes Jordão-Basso KC, de Souza Rastelli AN,de Andrade MF,et al.Effects of Photodynamic Therapy on the Adhesive Interface of Fiber Posts Cementation Protocols.JEndod.2018;44:173-178.

4. Victorino KR, Kuga MC, Duarte MA, Cavenago BC, Só MV, Pereira JR.The effects of chlorhexidine and ethanol on push out bond strength of fiber posts.JConserv Dent.2016;19:96-100.

5. Lorenzoni E Silva F, Pamato S, Kuga MC, Só MV, Pereira JR. Bond strength of adhesive resin cement with different adhesive systems. J ClinExpDent. 2017;9:e96-e100.

6. PortoI CCM, Nascimento TG, Oliveira JMS, Freitas PH, Haimeur A,França R. Use of polyphenols as a strategy to prevent bond degradation in the dentin-resin interface.Eur JOralSci.2018;126:146-158.

7. Sofan E, Sofan A, Palaia G,Tenore G, Romeo U, Migliau G. Classification review of dental adhesive systems: from the IV generation to the universal type.AnnStomatol.2017;8:1-17.

8. Silva e Souza MH Jr, Carneiro KG, Lobato MF, Silva e Souza Pde A, deGóesMF.Adhesive systems: important aspects related to their composition and clinical use.J Appl OralSci.2010;18:207-14.

9. Hewlett ER. Resin adhesion to enamel and dentin: a review. J Calif DentAssoc.2003;31:469-76.

10. Perdigão J. New concepts in dental adhesion. Northwest Dent.2000;79:29-33.

11. Van Meerbeek B, Perdigão J, Lambrechts P, Vanherle G. The clinical performance of adhesives. J Dent.1998;26:1-20.
12. Rosa WL, Piva E, Silva AF. Bond strength of universal adhesives: A systematic review and meta-analysis. JDent.2015l;43:765-76.

13. GianniniM,MakishiP,AyresAP,VermelhoPM,FronzaBM,NikaidoT,etal.Self-etch adhesive systems: a literaturere view.BrazDentJ.2015;26:3-10.

14. Alonso JRL, Basso FG, Scheffel DLS, de-Souza-Costa CA, Hebling J.Effect of crosslinkers on bond strength stability of fiber posts to root canal dentin and in situ proteolytic activity. J Prosthet Dent. 2018;119:494.e1-494.e9.

15. SpencerP,YeQ,ParkJ,ToppEM,MisraA,MarangosO,etal. Adhesive/Dentin interface: the weak link in the composite restoration. Ann Biomed Eng.2010; 38:1989-2003.

16. Nakabayashi N, Kojima K, Masuhara E. The promotion of adhesion by thein filtration of monomers in to tooth substrates. J biomed Mater Res.1982;16:265-73.

17. Van Noort R, Noroozi S, HowardI C, Cardew G. Acritique of bond strength measurements. J Dent. 1989; 17:61-7.

18. Carrilho MR, Tay FR, Donnelly AM, Agee KA, Tjäderhane L, Mazzoni A, etal. Host-derived loss of dentin matrix stiffness associated with solubilization of collagen. J Biomed Mater Res B Appl Biomater.2009;90:373-80.

19. TjäderhaneL, Nascimento FD, BreschiL, Mazzoni A,TersarioII L, Geraldeli S,et al.Strategiest opreventhy drolytic degradation of the hybrid layer- Are view. Dent Mater.2013;29:999-1011.

20. Wang Y, Spencer P. Hybridization efficiency of the adhesive/dentin interface with wet bonding. J DentRes. 2003;82:141-5.

21. Carvalho RM, Manso AP, Geraldeli S, Tay FR, Pashley DH. Durability of bond sand clinical success of adhesive restorations. Dent Mater.2012;28:72-86.

22. Mazzoni A,Papa V, Nato F, Carrilho M, Tjäderhane L, Ruggeri A Jr,et al. Immuno histochemical and biochemical as say of MMP-3 inhumandentine. JDent.2011;39:231-7.

23. Bedran-Russo AK, Pashley DH, Agee K, Drummond JL, Miescke KJ. Changes instiffness of demineralized dentin following application of collagen crosslinkers. Jbiomed Mater Res B Appl Biomater.2008;86:330-4.

24. Bedran-Russo AK, Yoo KJ, Ema KC, Pashley DH. Mechanical propertiesoftannic-acid-treateddentinmatrix. JDent Res.2009;88:807-11.

25. Al-Ammar A, Drummond JL, Bedran-Russo AK. The use of collagen cross-linking agents to enhance dentin bond strength. J Biomed Mater Res BApplBiomater.2009;91:419-24.

26. Castellan CS, Bedran-Russo AK, Karol S, Pereira PN. Long-term stabilityof dentin matrix following treatment with various natural collagen cross-linkers. J MechBehavBiomed Mater. 2011;4:1343-50.

27. Bedran Russo AK, Pauli GF, Chen SN, McAlpine J, Castellan CS, Phansalkar RS, et al. Dentin biomodification: strategies,renew able resources and clinical applications.Dent Mater.2014;30:62-76.

28. Castellan CS, Bedran-Russo AK, Antunes A, Pereira PN. Effect of dentin biomodification using naturally derived collagen cross-linkers: one-year bond strength study. IntJ Dent.2013;2013:918010.

29. Madhavan K, Belchenko D, Motta A, Tan W. Evaluation of composition and crosslinking effects on collagen-based composite constructs. ActaBiomater2010;6:1413–22.

30. Wang XH, Li DP, Wang WJ, Feng QL, Cui FZ, Xu YX, et al. Crosslinked collagen/chitosan matrix for artificial livers. Biomaterials 2003;24:3213–20.

31. Shrestha A, Friedman S, Kishen A. Photodynamically crosslinked and chitosan-Incorporated dentin collagen. JDentRes2011;90:1346–51.

32. Fawzy AS, Nitisusanta Lqbal K, Daood U, Beng LT, Neo J.Chitosan/Riboflavin-modified demineralized dentin as apotential substrate for bonding.JMechBehavBiomedMater 2013;17:278–89.

33. Kishen A, Shrestha S, Shrestha A, Cheng C, Goh C. Characterizing the collagen stabilizing effect of crosslinked chitosan nanoparticles against collagenase degradation.Dent Mater.2016Aug;32(8):968-77.

34. Santos J, Carrilho M, Tervahartiala T, Sorsa T, Breschi L, Mazzoni A, etal..Determination of matrix metalloproteinases in human radicular dentin.J Endod.2009;35:686-9.

35. Bandeca MC, Kuga MC, Diniz AC, Jordão-Basso KC, Tonetto MR. Effects of the Residues from the Endodontic Sealers on the Longevity of Esthetic Restorations.J ContempDentPract.2016;17:615-7.

4 CONCLUSÃO

A solução de tanino a 5% não proporciona maior longevidade da interface adesiva entre a dentina do espaço para pino e o sistema de cimentação com adesivo condiciona-e-lava e cimento resinoso convencional.

O uso de hipoclorito de cálcio a 6% para remover 0,01% de azul de metileno após PDT favorece a resistência de união do cimento resinoso autoadesivo (RelyX U200) e cimento de ionômero de vidro convencional (GC Gold Label 1 Luting & Lining Cement) para cimentação de pino de fibra de vidro.

O ácido peracético a 1% e o QMix apresentaram os maiores valores de resistência de união, enquanto o NaOCl 2,5% apresentou os menores valores. Os padrões de falha foram variados, com altas falhas adesivas na interface dentina/cimento para o tratamento da superfície radicular com NaOCl a 2,5% e Smear Clear. A penetração do cimento nos túbulos dentinários não foi afetada pela solução utilizada para enxaguar o espaço do pino.

REFERÊNCIAS*

1. Aranda-Garcia AJ, Kuga MC, Vitorino KR, Chávez-Andrade GM, Duarte MA, Bonetti-Filho I et al. Effect of the root canal final rinse protocols on the debris and smear layer removal and on the push-out strength of an epoxy-based sealer. Microsc Res Tec. 2003; 76: 533-7.

2. Magro MG, Kuga MC, Aranda-Garcia AJ, Victorino KR, Chávez-Andrade GM, Faria G et al. Effectiveness of several solutions to prevent the formation of precipitate due to the interaction between sodium hypochlorite and chlorhexidine and its effect on bond strength of an epoxy-based sealer. Int Endod J. 2015; 48: 478-83.

3. SudsangiamS, van Noort R. Do dentin bond strength tests serve a useful purpose?. J Adhes Dent. 1999; 1(1):57-67.

4. Pashley DH, Tay FR, Yiu C, Hashimoto M, Breschi L, Carvalho RM et al. Collagen degradation by host-derived enzymes during aging. J Dent Res. 2004; 83: 216-21.

5. Lorenzetti CC, Bortolatto JF, Ramos ATPR, Shinohara AL, Saad JRC, Kuga MC. The effectiveness of glass ionomer cement as a fiber post cementation system in endodontically treated teeth. Micros Res Tec. 2019; 82:1191-7.

6. Belizário LG, Kuga MC, Castro-Núñez GM, Escalante-Otárola WG, Só MVR, Pereira JR. Effects of different peracetic acid formulations on post space radicular dentin. J Prosthet Dent. 2018; 120(1): 92-8.

7. Ramos ATPR, Garcia Belizário L, Venção AC, Fagundes Jordão-Basso KC, de Souza Rastelli AN, de Andrade MF et al. Effects of photodynamic therapy on the adhesive interface of fiber posts cementation protocols. J Endod. 2018; 44(1): 173-8.

8. Belizário LG, Kuga MC, Hungaro Duarte MA, Só MVR, Keine KC, Pereira JR. Effect of fiber post space irrigation with different peracetic acid formulations on the bond strength and penetration into the dentinal tubules of self-etching resin cement. J Prosthet Dent. 2019; 122: 46-7.

9. Alencar CM, Verbicário Dos Santos J, Jassé F, Dos Santos GO, Escalante-Otárola WG, Castro-Nuñez GM et al. Protocols for mechanical cleaning of the post space on the bond strength between root dentin and cementation system. Oper Dent. 2021; 46: 467-75.

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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/guias-de-normalizacaoatualizado.pdf

10. Malacarne J, Carvalho RM, de Goes MF, Svizero N, Pashley DH, Tay FR et al. Water sorption/solubility of dental adhesive resins. Dent Mater. 2006; 22: 973-80.

11. Seseogullari-Dirihan R, Mutluay MM, Pashley DH, Tezvergil-Mutluay A. Is the inactivation of dentin proteases by crosslinkers reversible?. Dent Mater. 2007; 33: 62-8.

12. Carrilho MR, Carvalho RM, de Goes MF, di Hipólito V, Geraldeli S, Tay FR et al. Chlorhexidine preserves dentin bond in vitro. J Dent Res. 2007; 86: 90-4.

13. Aranda-Garcia AJ, Kuga MC, Vitorino KR, Chávez-Andrade GM, Duarte MA, Bonetti-Filho I ET AL. Effect of the root canal final rinse protocols on the debris and smear layer removal and on the push-out strength of an epoxy-based sealer. Micros Res Tech. 2006; 76: 533-7.

14. Magro MG, Kuga MC, Aranda-Garcia AJ, Victorino KR, Chávez-Andrade GM, Faria G et al. Effectiveness of several solutions to prevent the formation of precipitate due to the interaction between sodium hypochlorite and chlorhexidine and its effect on bond strength of an epoxy-based sealer. Inter Endod J. 2015; 48: 478-83.

15. Sudsangiam S, Noort R. Do dentin bond strength tests serve a useful purpose?. J Adhes Dent. 1999; 1: 57-67.

16. Pashley DH, Tay FR, Yiu C, Hashimoto M, Breschi L, Carvalho RM et al. Collagen degradation by host-derived enzymes during aging. J Dent Res. 2004; 83: 216-21.

17. Lorenzetti CC, Bortolatto JF, Ramos ATPR, Shinohara AL, Saad JRC, Kuga MC. The effectiveness of glass ionomer cement as a fiber post cementation system in endodontically treated teeth. Micros Res Tec. 2019; 82:1191-7.

18. Belizário LG, Kuga MC, Castro-Núñez GM, Escalante-Otárola WG, Só MVR, Pereira JR. Effects of different peracetic acid formulations on post space radicular dentin. J Prosthet Dent. 2018; 120(1):92-8.

19. Ramos ATPR, Garcia Belizário L, Venção AC, Fagundes Jordão-Basso KC, de Souza Rastelli AN, de Andrade MF et al. Effects of photodynamic therapy on the adhesive interface of fiber posts cementation protocols. J Endod. 2018; 44: 173-8.

20. Belizário LG, Kuga MC, Hungaro Duarte MA, Só MVR, Keine KC et al. Effect of fiber post space irrigation with different peracetic acid formulations on the bond strength and penetration into the dentinal tubules of self-etching resin cement. J Prosthet Dent. 2018; 122: 46-7.