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Renan Vaz Machry

INFLUÊNCIA DO TRATAMENTO DE SUPERFÍCIE EM PINOS ANATÔMICOS NA RESISTÊNCIA DE UNIÃO AO CIMENTO RESINOSO AUTOADESIVO

Dissertação de Mestrado

Santa Maria, RS

Renan Vaz Machry

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Dissertação apresentada ao Curso de Mestrado do Programa de Pós-Graduação em Ciências Odontológicas, Área de Concentração em Odontologia, ênfase em Prótese Dentária, da Universidade Federal de Santa Maria (UFSM, RS) como requisito parcial para obtenção do grau de **Mestre em Ciências Odontológicas**.

Orientador: Prof. Dr. Osvaldo Bazzan Kaizer

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Aprovado em 18 de julho de 2018:

Osvaldo Bazzan Kaizer, Dr. (UFSM)
(Presidente da banca/Orientador)

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Dedico este trabalho

Especialmente a minha família que me apoia em todos os momentos da minha caminhada e nas decisões que tomo. Nada seria possível sem uma base forte de amor, carinho e união. Tudo que conquisto é de todos e reflexo da educação e dedicação que recebi. Todos são importantes para mim e fazem parte da conclusão de mais essa etapa.

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RESUMO

INFLUÊNCIA DO TRATAMENTO DE SUPERFÍCIE EM PINOS ANATÔMICOS NA RESISTÊNCIA DE UNIÃO AO CIMENTO RESINOSO AUTOADESIVO

Autor: Renan Vaz Machry Orientador: Prof. Dr. Osvaldo Bazzan Kaizer

O objetivo do estudo é verificar a influência do tratamento de superficie e silanização de pinos anatômicos na resistência de união a um cimento resinoso autoadesivo. Para o teste de pushout, 80 dentes bovinos foram tratados endodonticamente e, após 24 horas, os preparos para os pinos foram realizados e as raízes foram alargadas com o auxílio de pontas diamantadas. pinos de fibra de vidro reembasados com resina composta microhíbrida, obtidos através da modelagem dos canais radiculares ampliados, foram divididos em grupos de acordo com o tratamento superficial aplicado (n=10): ausência de tratamento (GC), ausência de tratamento com aplicação de silano (GCS), jateamento com óxido de alumínio sem (GS) e com aplicação de silano (GSS), tratamento com peróxido de hidrogênio 35% sem (GP) e com aplicação de silano (GPS) e tratamento com ácido fluorídrico 10% sem (GF) e com aplicação de silano (GFS). Os pinos foram cimentados nos canais radiculares e, após 24 horas de armazenamento, fatias de 1,5mm de espessura foram obtidas para o teste de push-out. Os valores da resistência de união foram obtidos em MPa e as falhas foram classificadas através de estereomicroscópio em adesivas ou coesivas sendo apenas as falhas adesivas entre cimento resinoso e resina composta consideradas para análise estatística. Para o teste de microtração, foram confeccionados blocos de resina composta microhíbrida. A cada dois blocos, os mesmos grupos de tratamentos superficiais do teste de push-out eram aleatoriamente distribuídos para serem aplicados em uma das faces dos dois blocos. Estes foram cimentados um ao outro com cimento resinoso autoadesivo. Os 10 conjuntos de cada grupo obtidos foram levados à máquina de corte para confecção de 16 palitos por bloco para o ensaio de microtração. Metade dos corpos-de-prova de cada bloco foram imediatamente testados, e outra metade foi submetida à 12 mil ciclos de termociclagem e armazenamento por 120 dias em estufa antes do ensaio mecânico (n=80). Os dados foram tabelados em MPa considerando a área da interface adesiva e a carga necessária para separar os palitos. As falhas foram classificadas em adesivas ou coesivas. Para o teste de *push-out*, não houve diferença entre os grupos. Já no teste de microtração, para os espécimes imediatamente testados, obteve-se valores superiores de resistência de união para os blocos jateados seguidos da aplicação de silano. Por outro lado, a utilização de ácido fluorídrico 10% sem silano e peróxido de hidrogênio 35% com silano mostraram resultados significativamente inferiores aos demais tratamentos quando comparados àqueles sob mesma condição de aplicação ou não do agente de ligação química. A termociclagem causou queda estatisticamente significante nas médias de resistência de união para todos os grupos, além disso, foi possível observar valores superiores nos grupos jateados sem que houvesse diferença entre eles quanto a aplicação de silano. Uma amostra de cada grupo foi confeccionada para análise da superfície em microscopia eletrônica de varredura, que apresentou diferenças visuais de rugosidade superficial entre os grupos. Os resultados do teste de microtração permitem concluir que o jateamento da superfície de resina composta gera aumento na resistência de união com o cimento resinoso autoadesivo. No entanto, a interface que apresentou maior quantidade de falhas no teste de *push-out* foi entre cimento resinoso e dentina radicular.

Palavras-chave: Canais Radiculares Fragilizados. Cimento Resinoso Autoadesivo. Pinos Anatômicos. Pinos de Fibra. Pino de Fibra Reembasado. Raízes Alargadas. Resina Composta.

ABSTRACT

INFLUENCE OF SURFACE TREATMENT IN ANATOMIC POSTS ON THE BOND STRENGTH TO SELF-ADHESIVE RESIN CEMENT

Author: Renan Vaz Machry Advisor: Prof. Dr. Osvaldo Bazzan Kaizer

The aim of the study was to verify the influence of surface treatments on the bond strength of anatomic posts to a self-adhesive resin cement. For the push-out test, 80 bovine teeth were treated endodontically. After 24 hours, the preparation of the post space was performed and the roots were widened with diamond burs. Afterwards, relined glass fiber post with microhybrid composite resin (anatomic posts) obtained through the modeling of root canals were divided into groups according to the applied surface treatment (n = 10): absence of treatment (GC), absence of treatment with silane (GCS), sandblasting with aluminum oxide without (GS) and with silane application (GSS), treatment with 35% hydrogen peroxide without (GP) and with application silane (GPS) and treatment with 10% hydrofluoric acid without (GF) and with silane application (GFS). The posts were cemented to the root canals and, after 24 hours of storage, 1.5mm thick slices were obtained for the push-out test. The bond strength values were obtained in MPa and the failures were classified with a stereomicroscope in adhesive or cohesive, with only the adhesive failure between resin cement and composite resin were considered for statistical analysis. For the microtensile bond strength test (MTBS), were made blocks of microhybrid composite resin. The same surface treatments of the push-out test were randomly distributed to be applied on one of the faces of the blocks. Two blocks with same treatment were cemented together with self-adhesive resin cement. Ten sets of each group were obtained and cutting in a cut machine to obtain 16 sticks per block for the MTBS. Half of the specimens was immediately tested, and another half was subjected to 12,000 cycles of thermocycling and storage for 120 days before the mechanical test (n = 80). The data were tabulated in MPa considering the area of the adhesive interface and the load required to separate the sticks. The failures were classified as adhesive or cohesive. For the push-out test, there was no difference between the groups. In the MTBS the higher bond strength values was obtained for the sandblasted blocks followed by the silane application when in the specimens immediately tested. On the other hand, the use of 10% hydrofluoric acid without silane and 35% hydrogen peroxide with silane showed results significantly lower than the other treatments when compared to those under the same application condition or not the chemical coupling agent. The thermocycling caused a statistically significant decrease in the means of bond strength for all groups, in addition, it was possible to observe higher values in the sandblasted groups without there being any difference with silane application. A sample of each group was prepared for analysis of the surface by scanning electron microscopy, which presented visible differences of surface roughness between the groups. The MTBS' findings allow to conclude that the sandblasting of the composite resin surface generates an increase in bond strength with the self-adhesive resin cement. However, the interface that presented the greatest number of failures in the push-out test was between resin cement and root dentin.

Key words: Anatomical Post. Composite Resin. Fiber Post. Flared Roots. Relined Fiber Post. Self-adhesive Resin Cement. Weakened Root Canals.

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1 INTRODUÇÃO

A restauração protética de um dente tratado endodonticamente com destruição coronária extensa consiste em um desafio para o cirurgião-dentista e a necessidade de obtenção de retenção adicional para sua restauração com um sistema de retentores intrarradiculares vem sendo pesquisada há muitas décadas (MORGANO, 1996). Muito se evoluiu, mas ainda hoje um sistema de retentor e núcleo é geralmente inserido para restaurar dentes após o tratamento endodôntico (TRABERT; COONY JF, 1984; COLMAN, 1979). O objetivo principal é fornecer retenção para a porção coronária do núcleo de preenchimento e, consequentemente, suporte para a prótese ou restauração coronária (BARABAN, 1988).

O problema se intensifica quando o canal radicular apresenta anatomia oval ou foi ampliado além do habitual por conta de retratamentos endodônticos, reabsorção interna e cáries. Esta situação pode levar a falhas por deslocamento dos pinos resultantes da má adaptação dos retentores ao canal, problemas em função da espessura de cimento excessiva como ocorrência de bolhas e contração irregular na polimerização, além de maior risco de fraturas catastróficas devido à fina espessura das paredes radiculares (D'ARCANGELO et al., 2007; ZOGHEIB et al., 2008; WANDSCHER et al., 2014).

Atualmente, já está bem estabelecido na literatura que tanto o uso de núcleos metálicos fundidos quanto de núcleos de preenchimento com pinos de fibra apresentam resultados clínicos favoráveis quanto à longevidade, sendo as principais escolhas dos cirurgiões-dentistas (MARCHIONATTI et al., 2017). Entretanto, ainda que a forma de confecção de núcleos metálicos fundidos permita uma melhor adaptação aos canais radiculares com anatomia não uniforme, por uma questão de biomecânica é indicado que o sistema selecionado apresente pouca transmissão de tensões para a raiz dentária (DE CASTRO-ALBUQUERQUE et al., 2003; LANZA et al., 2005). Assim sendo, o alto módulo de elasticidade dos núcleos metálicos fundidos contraindica seu uso em dentes com conduto excessivamente alargado (WANDSCHER et al., 2014). Por conseguinte, seriam mais indicados pinos de fibra, que possuem módulo de elasticidade similar ao da dentina, promovendo melhor distribuição das tensões na raiz e a uma menor probabilidade de fraturas radiculares catastróficas (COELHO et al., 2009; SANTOS et al., 2010).

A fim de possibilitar melhor adaptação dos pinos de fibra pré-fabricados à anatomia não-uniforme de canais alargados ou com forma oval, desenvolveu-se a confecção de pinos anatômicos, ou seja, pinos de fibra de vidro reembasados no conduto radicular com resina composta (GRANDINI; SAPIO; SIMONETTI, 2003). Essa técnica reduz consideravelmente

a espessura de cimento resinoso necessário para preencher o espaço entre o retentor intrarradicular e as paredes do canal radicular, com isso, diminui a ocorrência de bolhas e falhas pela contração de polimerização do cimento resinoso e, consequentemente, melhora a união adesiva do pino cimentado ao dente (D'ARCANGELO et al., 2007; MACEDO; FARIA E SILVA; MARTINS, 2010; FARINA et al., 2016, ROCHA et al., 2017).

Em uma condição atual de canais não fragilizados, a maioria das falhas com pinos de fibras pré-fabricados é, geralmente, pela descimentação dos mesmos, porém preservando a estrutura dentária (MARCHIONATTI et al., 2017). Nesse ponto, atualmente os cimentos resinosos autoadesivos apresentam apresentarem resultados favoráveis na adesão de pinos de fibra de vidro (SARKIS-ONOFRE et al., 2014), além de uma técnica menos sensível por eliminar passos prévios à cimentação como o pré-tratamento do canal radicular e por apresentar método simplificado de aplicação do cimento resinoso no interior do conduto (SKUPIEN et al., 2015).

Quando cimentos resinosos autoadesivos foram testados junto à técnica de pinos anatômicos, foi possível obter melhores resultados quanto à adesão do conjunto ao canal radicular se comparado a cimentos resinosos convencionais (DA SILVEIRA-PEDROSA et al., 2016; DE SOUZA et al., 2016). Algumas técnicas de tratamento de superfície de blocos de resina composta, como jateamento com óxido de alumínio e aplicação de ácido fluorídrico em diferentes concentrações foram testadas previamente à cimentação dessas peças com cimento resinoso autoadesivo para verificar uma possível melhoria da adesão (HARORLI et al., 2015). Entretanto, ainda há necessidade de novos estudos na literatura sobre o tratamento superficial de pinos anatômicos.

Nesse contexto, devemos considerar a presença da interface entre o cimento resinoso e a resina composta, ou seja, a adesão entre diferentes compósitos. O jateamento com óxido de alumínio foi testado sobre superfícies de restaurações de resina composta com necessidade de reparo por criar uma área com maior rugosidade superficial, aumentar a energia de superfície e expor componentes de carga da resina composta (RATHKE; TYMINA; HALLER, 2009, BAENA et al., 2015; LOOMANS et al., 2016). Como resultado pode-se observar aumento na união do material existente com o novo compósito adicionado. força de (RODRIGUES; FERRACANE; DELLA BONA, 2009; CHO et al., 2013; NASSOOHI et al. 2015, SOUZA et al. 2017). Assim como jateamento, o peróxido de hidrogênio promove aumento da rugosidade de restaurações de resina composta presentes em boca quando aplicado com finalidade clareadora, (ATTIN et al., 2004). Seu uso, no entanto, não foi testado sobre superfícies de pinos anatômicos com finalidade de aumentar a força de união a outro compósito.

O ácido hidrofluorídrico é indicado para condicionamento superficial de materiais cerâmicos (VENTURINI et al., 2015), entretanto vem sendo testado sobre superfícies de resina composta por também aumentar a rugosidade e a energia de superfície desses compósitos (ÖZCAN et al., 2003; GUPTA et al., 2015). Todavia, acredita-se que possa haver enfraquecimento e aceleração da degradação hidrolítica da resina composta por conta da penetração de água nos espaços entre as partículas de carga da resina condicionada e, consequentemente, desorganização da camada de silano responsável pela estabilidade entre matriz e carga (RODRIGUES; FERRACANE; DELLA BONA, 2009). No entanto, desconhecemos se há na literatura estudos quanto sua aplicação sobre pinos anatômicos.

O silano, além de ser parte da composição das resinas compostas, é indicado também como agente de ligação química entre partículas orgânicas e inorgânicas que estão presentes em materiais resinosos (JUNG; MATINLINNA, 2012). Sua aplicação é recomendada no reparo de restaurações de resina composta, sobretudo após tratamentos de superfície como o jateamento com óxido de alumínio, pois interage com as partículas de carga da resina composta (BOUSCHLICHER; REINHARDT; VARGAS, 1997; TEZVERGIL; LASSILA; VALLITU, 2003). Seus benefícios são consideráveis quando usados sobre pinos de fibra (SKUPIEN et al., 2015), mas sua aplicação sobre resina composta segue sendo questionada (RATHKE et al., 2009) e, portanto, sua aplicação merece ser ainda mais explorada.

Visando uma evolução da técnica de cimentação de pinos anatômicos e minimizar o risco de falhas, é importante testar as principais variáveis deste procedimento. Por isso, são necessários estudos que esclareçam que tipo de tratamento de superfície da resina composta da superfície do pino anatômico promove melhor resistência de união ao cimento resinoso autoadesivo.

2 ARTIGO - INFLUENCE OF SURFACE TREATMENT IN ANATOMIC POSTS ON THE BOND STRENGTH TO SELF-ADHESIVE RESIN CEMENT

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INFLUENCE OF SURFACE TREATMENT IN ANATOMIC POSTS ON THE BOND STRENGTH TO SELF-ADHESIVE RESIN CEMENT

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INFLUENCE OF SURFACE TREATMENT IN ANATOMIC POSTS ON THE BOND STRENGTH TO SELF-ADHESIVE RESIN CEMENT

Short Title: Bond Strength of relined fiber posts cemented with RelyX U200

Clinical significance:

When luting relined fiber posts with self-adhesive cement RelyX U200, the surface treatment of the posts played no role in the adhesion of the fiber posts to root dentine, in the push out test. However, the microtensile test showed with the sandblasting increase the bond strength of composite resin to self-adhesive resin cement. On the other hand, after thermal aging the silane coupling agent application promoted an increase on the adhesion in control and hydrofluoric acid groups, but did not promote same benefit to this sandblasted resin surface.

Abstract.

Purpose: The aim of this study was to evaluate the effect of composite resin surface treatment and silanization on bond strength of relined fiber posts cemented with self-adhesive resin cement.

Methods and Materials: Two mechanical tests were applied in this study: push-out and microtensile test (MTBS) where 80 single-rooted bovine teeth and 1280 microbars were respectively used. Endodontic treatment was performed on the push-out teeth. After that, they were fragilized with a diamond bur, the glass fiber posts were adapted with composite resin (Filtek Z250, 3M ESPE) to the root canals, received the surface conditioning and were cemented with self-adhesive resin cement (RelyX U200, 3M ESPE). Four slices per tooth were obtained to the mechanical test. For the MTBS, the microbars (n=80) was also subdivided into two aging cycling conditions (immediate test or 12,000 thermocycling cycles plus 120 days of storage). Previously, 160 blocks of composite resin (Filtek Z250, 3M ESPE) were cemented to each other belonging to the same treatment group with self-adhesive resin cement (RelyX U200, 3M ESPE). The 80 sets (10 per surface treatment group) were then cut into the microbars (16 per set): 8 were immediately tested while the other 8 were thermocycled and stored prior to mechanical testing. The failure mode was classified in the two mechanical tests and micrographic surface analyzes were performed on each surface treatment of relined posts. Finally, the data were subjected to statistical analysis

Results: There was no statistically significant difference in the results of the push-out test. In MTBS, the surface treatment had a significant effect in all aging and silanization conditions (p = 0.000). The thermocycling decrease the bond strength for all groups. However, the sandblasting presented the superior bond strength average compared to the others groups after thermal aging and no difference was present when the silane coupling agent was applied on these sandblasted specimens.

Conclusions: The bond strength of relined posts cemented with self-adhesive resin cement appears to be more critical at the interface between cement and root dentin. However, the surface treatment of the composite resin with aluminum oxide sandblasting improves the bond strength durability to self-adhesive resin cement.

Key words: Anatomical Post. Composite Resin. Fiber Post. Flared Roots. Relined Fiber Post. Self-adhesive Resin Cement. Weakened Root Canals.

INTRODUCTION

The restoration of teeth with extensive coronary destruction usually requires the use of retainers which provide core retention and consequently support for prosthesis or coronary restoration^{1,2}. The problem intensifies when the root canal presents oval anatomy or was greatly enlarged by endodontic treatments, internal resorption and caries; leading to critical situations such as displacement of the posts resulting from poor adaptation to the root canal, problems caused by excessive cement thickness (voids and irregular contraction of polymerization) and risk of catastrophic fractures due to the thin thickness of the root walls³⁻⁵.

Although cast post-and-cores and fiber posts present equally favorable clinical results regarding longevity in teeth with regular root canal⁶, the high modulus of elasticity contraindicates its use in teeth with excessively wide conduit⁵, while fiber posts seem more indicated in this case because they have modulus of elasticity similar to that of the dentin, promoting a better distribution of the tensions in the root and a less probability of catastrophic root fractures^{7,8}.

In order to allow a better adaptation of prefabricated fiber posts to the non-uniform anatomy, flared or oval-shaped canals, relined posts were developed, that is fiberglass posts relined in the root canal with composite resin⁹. This technique considerably reduces the thickness of resin cement required to fill the space between the retainer and the root canal walls, reduces the occurrence of voids and failure by the polymerization contraction of the resin cement and, consequently, improves the adhesive bonding of the post to the root canal^{3,10-12}.

The self-adhesive resin cements are options that present a less sensitive technique to eliminate pre-cementation steps and to present a simplified method of application of the resin cement inside the root canal¹³. Moreover, when tested with the relined posts, it was possible to obtain better results regarding the adhesion of the whole to the root canal when compared to conventional resin cements^{14,15}.

Some techniques of surface treatment of composite resin blocks, such as aluminum oxide blasting and hydrofluoric acid etching have been previously tested to cement these blocks with self-adhesive resin cement to verify a possible improvement of adhesion¹⁶. These treatments seek to create an area with greater surface roughness, increase surface energy and expose composite resin filler components¹⁷⁻²¹. In the same way, hydrogen peroxide promotes increased roughness of composite resin restorations present in the mouth when applied with a bleaching purpose²². Up to present, it is unknow studies evaluating the use of these surface treatments on relined posts cemented with self-adhesive resin cement to the root canal.

The silane, besides being part of the composition of the composite resins, is also indicated as coupling agent between organic and inorganic particles that are present in resinous materials²³. Its application is recommended in the repair of composite resin restorations, especially after surface treatments such as sandblasting with aluminum oxide, as it interacts with the composite resin loading particles^{24,25}. However, besides its application in composites is still questioned¹⁸, its application on relined posts deserves to be explored.

Therefore, the aim of this present study was to evaluate the effect of different surface treatments and silane application on the bond strength durability of relined posts cemented with self-adhesive resin cement to the root canals. The null hypothesis is, therefore, that these surface treatments and silane application would have no influence on the bond strength of the relined posts.

METHOD AND MATERIALS

Experimental design

Two mechanical tests were applied in this study: push-out and microtensile test.

For the push-out test, the sample size was calculated using the OpenEpi 3.01 considering parameters that were based on a previous pilot study considering a power of 80% and significance level was 0.05. Eight bovine teeth per group were necessary. However, because of the variability of the root anatomy of the bovine teeth, ten teeth per group were used in this study (n = 10).

For the microtensile bond strength test (MTBS), the microbars were used as sample unit. For this, 80 specimens per group were obtained from microhybrid composite resin blocks cemented together belonging both from the same experimental group.

For both tests, the specimens were randomly allocated into eight groups considering the absence of surface treatment and three possible conditions following silane application or not. Furthermore, for the MTBS, the specimens were also divided between immediately tested and submitted to thermocycling and storage before the test, and there were 16 groups for that part of the study (Table 1).

Tooth Selection and Preparation

Bovine incisors were obtained and sectioned to obtain roots with standard lengths of 16 mm. The roots were then selected according to the diameter of a size 80 K-file (Dentsply Maillefer, Ballaigues, Switzerland), in order to reduce the size variation between root canals. Apical root

portions were included in a chemically cured acrylic resin (VIPI, Pirassununga, Brazil) block. The specimens were fixed on a surveyor, with the long axes of the teeth and the resin block parallel to each other and perpendicular to the ground.

Endodontic Procedures

Canal patency was established with a size 15 K-file (Dentsply Maillefer). The working length was set at 1 mm from the apex. Root canals were prepared by using endodontics files (Dentsply Maillefer). Initially, the cervical portion of the roots was prepared by using gattesgliden drills (Dentsply Maillefer). Then, the step-back technique was applied. Each canal was irrigated with 2 mL of a 2.5% sodium hypochlorite (Novaderme, Santa Maria, Brazil) between each instrument change. Specimens were irrigated with a 5 mL of 17% EDTA (Novaderme) for tree minutes and subsequently rinsed with 2mL of distilled water. Next, they were dried using size 80 paper points (Dentsply Maillefer).

AH Plus (Dentsply Maillefer) was mixed according to the manufacturer's instructions and placed in working length by using a lentulo spiral (Dentsply Maillefer). Gutta-percha cones (Dentsply Maillefer) compatible with the diameter of the last instrument used for the instrumentation of the apical third of the root canal were used. The compression technique was cold lateral condensation with R8 accessory cones (Tanari, Manacapuru, Brazil). The excess of gutta-percha in the coronal portion was removed with a hot instrument. Roots were stored for 72 hours at 37°C and 100% humidity to allow the sealers to set.

Post Space Preparation

Root canal filling was partially removed using a hot instrument and sizes 1, 2, 3 and 4 Largo drills (Dentsply Malleifer). Post space preparation was completed using the Whitepost DC N2 (FGM, Joinville, Brazil) bur at 12mm. Then, the root canals were fragilized with diamond burs #4137 (KG Sorensen, Cotia, Brazil) in high rotation (Extra Torque 605C; Kavo, Joinville, Brazil), at 10 mm in the canal under irrigation with distilled water.

Posts Preparation

To obtain relined fiberglass posts, the Whitepost DC N2 (FGM, Joinville, SC, Brazil) were cleaned with 70% alcohol and was applied a silane coupling agent (RelyX Ceramic Primer; 3M, ESPE, São Paulo, Brazil) according to the manufacturer's instructions. The composite resin (Filtek Z250; 3M ESPE, São Paulo, Brazil) was condensed inside the previously lubricated root canal, the post was positioned, and the resin was light cured for 5 seconds

using a 1200Mw/cm² LED light-curing unit (Radii Cal; SDI, Melbourne, Australia) from the occlusal surface. The relined posts were removed from the canal, light-cured for 40 seconds, and reinserted to verify adaptation⁵. The relined posts were divided into the surface treatment and silane application groups (Table 1).

Specimens Production for MTBS

160 composite resin blocs (Filtek Z250; 3M-ESPE, São Paulo, SP, Brazil) were prepared using a silicon matrix (4 mm high and 8 mm sides) placed on a glass plate coated by polyester strip. Each increment (± 2 mm) was condensed using a #1 spatula (Golgran, São Caetano do Sul, Brazil) and photoactivated for 40 seconds (Radii Cal; SDI). The last layer was covered with a polyester strip and compressed using a glass slide to obtain a flat surface. The sample was photoactivated through the glass plate with the polyester strip in contact with the surface of the composite resin. The blocks obtained were divided into the surface treatment and silane application groups (Table 1).

Luting procedures

Previously at the cementation procedure and after all specimens were distributed in the different study groups (Table 1), they were washed for 10 seconds with distilled water spray and dried with water-oil-free spray. For those included in the silanization groups, the surfaces were cleaned with 70% alcohol, the silane agent was applied with a disposable microbrush (Cavibrush, FGM, Joinville, Brazil) rubbed for 5 seconds, with evaporation of solvent for 5 minutes.

RelyX U200 (3M ESPE, Seefeld, Germany) was mixed according to the manufacturer's specification, inserted into the root canal using a lentulo spiral (Dentsply Maillefer), and immediately after, the relined fiber post was inserted by manual pressure. The cement was light-cured for 40 seconds (Radii Cal; SDI) being 10 seconds on each face. Roots were stored for 24 hours at 37°C.

For MTBS blocks, the RelyX U200 (3M ESPE) was mixed according to the manufacturer's specification and applied on the surface of one of the blocks. Another block from the same group as the previous was immediately positioned on the first and a load of 2.5N was applied onto the assembly through a static press. The cement excesses were removed with microbrush (FGM, Joinville, Santa Catarina, Brazil) waits for 3 minutes for resin cement to settle. The photoactivation was performed for 25 seconds on the interface on

one side of the blocks. The load was removed and the assembly again light-cured for another 80 seconds (20 seconds each side).

Push-out test

The teeth were fixed on a metal base in the cutting machine (Isomet 1000 Precision Saw, Buehler, Warwick, UK) and then sectioned perpendicular to the long axis of the root. The first cervical slice (approximately 1 mm thick) was discarded, and four other slices per specimen (thickness: 1.5 ± 0.3 mm) were obtained (40 per group). Each slice was positioned on a metallic device with a central opening (\emptyset =3 mm) larger than the canal diameter. The most coronal portion of the specimen was placed downward.

The push-out test was performed in a universal testing machine (Emic DL-2000; Emic, São José dos Pinhais, Brazil) at a speed of 1mm/min. A metallic cylinder (Ø extremity=0.8 mm) induced a load on the post in an apical to coronal direction, without applying any pressure on the composite resin, cement and/or dentin.

Bond strength values (α) in MPa were obtained as follows: $\alpha = f/a$, where f = load for specimen rupture (N) and a = bonded area (mm²). To determine the bonded interface area, this formula was used: $A = 2\pi g.(R_1+R_2)$, where $\pi = 3.14$, g = slant height, $R_1 = smaller$ base radius, and $R_2 = larger$ base radius. To determine the slant height, the following calculation was used: $g^2 = (h2 + [R_2 - R_1]^2)$, where h = section height. R_1 and R_2 are obtained by measuring the internal diameters of the smaller and larger base, respectively, which corresponded to the internal diameter between the root canal walls. The diameters and h were measured using a digital caliper (Starret 727, Starrett, Itu, São Paulo, Brazil).

Microtensile Bond Strength Test (MTBS)

The blocks were sectioned into microbars with an interface area of about 1 mm × 1 mm × 8 mm, using a diamond disk at low speed, under water cooling (Isomet, Buehler, USA), producing a total of approximately 16 microbars each. Half was immediately subjected to the microtensile test (baseline) and the other half was thermocycled by 12,000 cycles between 5°C and 55°C with a residence time of 30 seconds and transfer time of 2 seconds (Nova Etica, São Paulo, Brazil) and stored in 37°C distilled water for 120 days.

Each sample was measured using a digital caliper (Starrett 727; Starrett, Itu, Brazil) and positioned in Geraldeli devices with cyanoacrylate glue (Three Bond Gel; Three Bond, Diadema, Brazil). The MTBS was applied in a universal test machine (EMIC DL-2000, São José dos Pinhais, Brazil) with a load cell of 50kN (force limit: 500N) at a speed of

0.5mm/min. The bond strength in MPa was calculated whereas the force required to cause failure (in Newton) and the area of the bonded interface (in millimeters): $\alpha = f/a$.

Failure mode analysis

After the push-out and microtensile tests, the specimens were analyzed at x10 magnification with a stereomicroscope (Zeiss Stemi SV6; Carl Zeiss, Jena, Germany). For push-out test the failure modes were categorized as follows: Ac/d = predominant adhesive at cement/dentin interface failure, Ac/cr = predominant adhesive at composite resin/cement interface failure, Cr/p = predominant adhesive at composite resin/fiber post interface failure, COE = predominant cohesive in some material or dentin. In MTBS the failure modes were categorized as adhesive (predominant at composite resin/cement interface failure) or cohesive (predominant in the composite resin or cement). Moreover, some glue failures were observed.

For the push-out test, only the Ac/cr failures were considered for statistical analysis because this is the interest interface. In MTBS, no adhesive failure (cohesive or glue failures) specimens were excluded from the study given that these types of failures did not represent real bond strength.

Scanning Electron Microscopy

One sample of relined fiber post from each group was prepared for surface analysis by scanning electron microscopy (VEGA3; TESCAN, Brno, Kohoutovice, Czech Republic) at x2000 magnifications to assess changes in surface topography.

Data analysis

The mean of bond strength distributions was checked with the Shapiro-Wilk test. The normal distribution of data was confirmed

In push-out test, two-way analysis of variance (IBM SPSS Software; IBM, New York, USA) were used for statistical analysis considering the surface treatment and the silane application. The significance level was set at 5%.

For MTBS, one-way analysis of variance (ANOVA) were used for statistical analysis to investigate ascertain was difference between the groups regarding the surface treatment within the same condition of silanization and/or thermocycling. The Student's t-test was applied to find averages differences within these indicated factors

RESULTS

Push-out test

According to Table 2, the effect of surface treatment and silanization on push-out bond strength were not statistically significant. In addition, Moreover, the number of failures in the interface of interest was lower than in the others, except in posts treated with hydrogen peroxide without silane application that presented 37.5% of failures between cement and composite resin.

Microtensile Bond Strength Test

The Table 3 demonstrates that all surface treatments without silanization were similar on baseline. The exception was the hydrofluoric acid group, which, in this context, presented the lowest average. The silanized baseline specimens presented other results (p-value = 0.000). In this case, sandblasting presented the highest mean (81 MPa \pm 11.4), followed by hydrofluoric and control acid groups (68.5 MPa \pm 11.7, 66.7 MPa \pm 11.8), which were not different from each other. The hydrogen peroxide group showed a marked decrease (48.7 MPa \pm 11.3). When comparing the means of the specimens immediately tested, the silanization increased the mean of the bond strength. Only the absence of surface treatment showed no statistically significant difference.

Analyzing the results of the specimens tested after thermocycling and storage, it was possible to observe that the sandblasting obtained the highest values of the bond strength to the others groups on the same aging and silanization conditions (p-value = 0.000). The specimens treated with hydrogen peroxide had inferior means also for the two conditions while the hydrofluoric acid and control groups were in intermediate position, not presenting any difference between them. In addition, the latter were the only ones that showed differences between silane and non-silane application.

Regarding to failures, it is possible to observe that there were more adhesive failures after thermocycling compared to the specimens immediately tested (Table 4), with exception for the hydrofluoric acid group without silane. In general, the sandblasted and untreated surfaced groups had fewer adhesive failures than the other two. The exception was the untreated group with silane application that after thermocycling presented a large increase in the number of adhesive failures. Although not considered for statistical tests, some specimens showed glue failures used in the test device and were also classified in Table 4. Pre-test failures were not added, therefore not all groups present a total of 80 specimens.

Scanning Electron Microscopy Images

The images (Figure 1) showed the difference of the surface of the composite resin after each surface treatment and application of the silane agent. The topography of the treated surfaces was modified by acid conditioning (hydrofluoric acid) and sandblasting with removal of the matrix of the composite resin, opening the spaces in nanoscale, resulting in a relatively rough surface and with exposure of charge particles. A significant difference in surface roughness pattern can be observed when comparing specimens without application of silane and silanized specimens.

In the surfaces treated with hydrofluoric acid it is possible to observe gaps caused by the hydrolytic dissolution while on the sandblasted surfaces the pattern of roughness appears more uniform. In the hydrogen peroxide group, it is not possible to observe differences compared to the untreated surface group.

DISCUSSION

This study showed significant differences in bond strength to microtesile test (MTBS) between self-adhesive resin cement and composite resin when different surface treatments were applied, therefore, the null hypothesis was rejected. Although no statistical differences were noted in the push-out test.

The use of bovine teeth in adhesion studies is well accepted due to the similarities between human teeth on previous findings²⁶. However, the adhesion tests of the root canal have adhesives failures, mainly in the interface between the resin cement and the dentin, being the most critical area^{11,12}. Although the methodology of the push-out test simulates clinical conditions of luting posts in root canals, the results of bond strength between materials are not the most accurate because the test induces frictional force beyond bond strength²⁷.

The microtensile bond strength test (MTBS) isolates the interest interface from the study generating a more homogeneous tension distribution at the interface than other mechanical tests^{28,29}. Therefore, its results bring more specific interpretations of the adhesive resistance between the resin cement and composite resin. Besides that, the results obtained may even extrapolate to wider interpretations. In this methodological outline was possible to observe significant differences between the groups tested.

The cohesive failures of MTBS occur because the composite resin has tensile strength around 65.95 MPa (28.1 - 102.1 MPa)³⁰. The adhesive failure rates obtained in the present

study are within this range, therefore, the groups that presented the most cohesive failure had this outcome due to the high adhesive resistance between the self-adhesive resin cement and the composite resin, which was greater than or equal to the fracture strength of the composite resin. Therefore, Palasuk et al. report that after sandblasting with aluminum oxide on silorane composite resins, it was produced microtensile bond strength not different from the cohesive strength of this material³¹. The same can be found from our findings, where we find values of bond strength between the composite resin and the self-adhesive resin cement used similar to the cohesive strength value of the methacrylate-based composite resin³⁰.

In general, the results of our study regarding the surface sandblasting of the specimens are in accordance with what has been presented in previous studies that suggest that there is an improvement in the bond strength of a composite to a new resinous material ^{19,21,32,33}. However, the studies carried out to date have evaluated this adhesion in aged composite resin. What we can observe is that the adhesion benefit of increased roughness through sandblasting is also significant in composite resins that did not go through the aging process like those used in relined posts.

Scanning electron microscopy (SEM) evidenced the significant increase of surface roughness in specimens sandblasted with 45 microns aluminum oxide particles in the same way as previous studies^{33,34}. Among the factors that influenced the adhesion of the self-adhesive resin cement to the prepared substrate, this seemed the most important in the bond strength between the two composites. The justification for this is that, although adhesion of a resinous material over another avid to receive new bonds seems predictable considering the composition of the materials, in the long term it is benefited by the increase of roughness by mechanical interlocking and exposure of the silica particles³⁴. In the baseline where this bond was not exposed to adverse environmental conditions, the results shows that it does not really seem necessary to apply mechanical or chemical treatment (silane) - the association between sandblasting and silanization. However, the greatest difference is observed after thermocycling when the materials were exposed to thermal cycling, which is known to cause contraction aging and structural expansion³⁵.

The literature reports that 10,000 cycles of thermocycling of composites correspond to one year of an exposure to the conditions of variation in an oral environment³⁶. The thermal cycle aging process attempts to reproduce the hydrolytic degradation in the resin matrix that occurs in the buccal environment³⁷. Therefore, MTBS results after thermocycling indicate the behavior of the materials in the long term. In this context, what the study finds as the main

finding is that sandblasting generates greater bond strength, regardless of the application or not of the silane coupling agent.

The union between the silane and the composite resin deteriorates over time due to the hydrolysis, since the resins are permeable³⁹. It is believed that this deterioration will be less if surface preparation is adequate, providing micro-mechanical retention performed prior to silane treatment³⁹. However, contrary to what can be observed on dental ceramics⁴⁰, our study demonstrates that this effect did not occur on composite resin surfaces. After the thermocycling, the silane application was better in the control group, that is, without increase of roughness, and there was no difference in the blasted group, which is according to previous study that will show no benefit in associating sandblasting with silane application⁴¹.

In the same way as sandblasting with aluminum oxide, hydrofluoric acid promotes a great change in the topography of the composite when applied on a composite resin surface. However, during the conditioning process, there is water penetration and hydrolytic degradation, that is, breaking the silane bond between matrix and load and consequent weakening of the composite 17,42. Thus, the surface of the composite resin becomes rough, but the structure becomes very weak and prone to microcracks. The image of SEM evidences areas of loss of structure that corroborates this possibility.

Regarding hydrogen peroxide, it is considered a simple and effective agent for the treatment of methacrylate-based polymeric materials such as fiber posts⁴³. However, we find that this cannot be considered for surfaces of relined posts where this substrate is of composite resin. Generally, the inferior results were found in the groups with this treatment, and the SEM images showed no significant variation in the composite surface morphology when compared to the absence of treatment. Hydrogen peroxide triggers an oxidation process on the surface of composites. Thereafter, there is conversion into hydroxyl groups by a breakdown of the molecular bonds of the material⁴⁴. From this, the silane would react with the hydroxyl groups formed, improving the chemical bonding and the wettability for a new bond⁴⁵. However, although the literature shows that silane application could reverse the fall in hydrogen peroxide bond strength⁴⁶, it was not possible to observe this result in our study.

Some limitations of this current study can be depicted as follows: no aging condition (thermocycling) in the push-out specimens and the high failure rate between the different interfaces of interest in the study, as well as cohesive failures in the microtensile test. It is necessary that the bond strength between the self-adhesive resin cement and the root dentin be most investigate in order to obtain better results.

CONCLUSIONS

Based on the present results and within the limitations of this in vitro study, it can be concluded that:

- 1. The surface treatments and silanization have influence on the bond strength of composite resin to self-adhesive resin cement on the microtensile bond strength test;
- 2. After sandblasting the composite resin surface with aluminum oxide, the aged specimens present the greatest bond strength with the resin cement self-adhesive and the application of coupling agent silane no present adhesiveness durability influence on the microtensile bond strength test;
- 3. The push-out test did not result in statistical difference and showed that the main failure occurs between cement and root dentin.

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Figures

Figure 1 – Representative SEM image at 2000X magnification of relined fiber post surface after non-surface treatment, 35% Hydrogen Peroxide treatment, 10% Hydrofluoric Acid treatment and sandblast treatment: (A) non-silanized. (B) silanized.

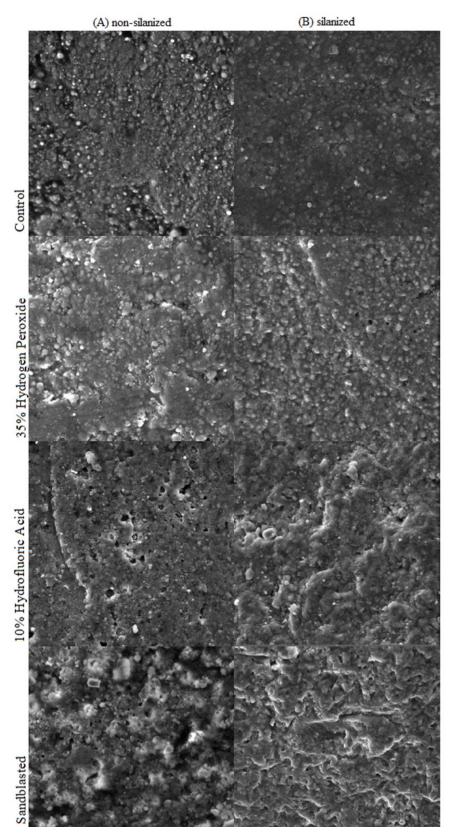


Table 1: Experimental groups regarding the surface treatment used, the application or not of the silane agent and thermocycling plus storage (only for microtensile test).

Surface Treatment	Name/Brand Protocol Application	Silane Application	Push- out groups	Thermocycling and storage*	MTBS Groups
Control	No surface treatment	NO	GC	NO (baseline) YES	GCi GCt
		YES	GCS	NO (baseline) YES	GCSi GCSt
10% Hydrofluoric	(Dentsply, Petrópolis, Brazil)	NO	GF	NO (baseline) YES	GFi GFt
Acid	Acid application for 60 seconds. ⁴¹	YES	GFS	NO (baseline) YES	GFSi GFSt
Hydrogen	(Whiteness HP; FGM, Joinville,	NO	GP	NO (baseline) YES	GPi GPt
Peroxide 35%	Brazil) Gel application for 60 seconds.	YES	GPS	NO (baseline) YES	GPSi GPSt
Sandblasting with 45µm Aluminum Oxide	(Polidental, Cotia, Brazil) 10 seconds at	NO	GS	NO (baseline) YES	GSi GSt
	distance of 5mm and pressure of 2.8	YES	GSS	NO (baseline)	GSSi
	bar. ⁴¹			YES	GSSt

^{*}Only the specimens of the microtensile test were divided for thermocycling and storage

Tables

Table 2: Mean of Bond Strength Values and Failure Modes Distribution After Push-Out Test

Groups	Bond strength	Failures				
		Ac/d	Ac/cr	Cr/p	COE	Total
GC	2.7± 1.8 a,A	27 (67,5%)	5 (12,5%)	7 (17,5%)	1 (2,5%)	40
GCS	$2.0 \pm 1.4 \text{ a,A}$	32 (80%)	3 (7,5%)	5 (1,25%)	0	40
GF	$2.5 \pm 1.4 \text{ a,A}$	27 (67,5%)	5 (12,5%)	8 (20%)	0	40
GFS	2.2 ±1.0 a,A	21 (52,5%)	7 (17,5%)	12 (30%)	0	40
GP	$2.6 \pm 2.7 \text{ a,A}$	10 (25%)	15 (37,5%)	15 (37,5%)	0	40
GPS	$1.6 \pm 0.3 \text{ a,A}$	27 (67,5%)	8 (20%)	5 (12,5%)	0	40
GS	2.2 a,A*	22 (55%)	2 (5%)	16 (40%)	0	40
GSS	$2.0 \pm 0.5 \text{ a,A}$	26 (65%)	6 (15%)	8 (20%)	0	40
	Total	192 (60%)	51 (15,94%)	76 (23,75%)	1 (0,31%)	320

Abbreviations: Ac/d: cement/dentin, Ac/cr: composite resin/cement, Cr/p: composite resin/fiber post, COE: cohesive failure in dentin.

Upper case letters compare the surface treatment factor under the same silanization condition Lowercase letters compare the silanization factor of the same surface treatment *no sufficient values were obtained to calculate the standard deviation

Table 3: Descriptive table of the means of the values in the microtensile test (MTBS) in MPa.

	Base	eline	Thermocycling			
Treatment	No silanization	Silanization	No silanization	Silanization		
No surface	63.6 ± 18.9 A,a*	66.7 ± 11.8 B,a*	42.4 ± 13.9 B,a°	52.3 ± 11.1 B,b°		
treatment						
Hydrofluoric	55.5 ± 11.0 B,a*	68.5 ± 11.7 B,b*	$45.9 \pm 10.8 \text{ B,a}^{\circ}$	$51.8 \pm 10.9 \text{ B,b}^{\circ}$		
Acid 10%						
Hydrogen	65.2 ± 12.4 A,a*	48.7 ± 11.3 C,b*	34.7 ± 9.1 C,a°	35.3 ± 8.3 C,a°		
Peroxide 35%						
Sandblasting	71.8 ± 16.3 A,a*	81 ± 11.4 A,b*	$60.6 \pm 12.7 \text{ A,a}^{\circ}$	57.7 ± 12.7 A,a°		
<i>p</i> -value	0.000	0.000	0.000	0.000		

Different lowercase letters indicate statistical difference between silane application within the same treatment and aging condition.

Different upper-case letters indicate statistical difference between treatments on same silanization and aging condition.

Different symbols (*/°) indicate statistical difference between the means of the specimens tested immediately and those submitted to thermocycling on same silanization condition.

Table 4: Descriptive table of failures modes in each group on MTBS

	ADHESIVE		COESIVE		Glue fail		TOTAL	
	Baseline	Thermo	Baseline	Thermo	Baseline	Thermo	Baseline	Thermo
	29	39 (48,8)	46 (57,5)	41 (51,3)	5 (6,25)	0	80	80
GC	(36,25)							
CCS	29	56 (70,9)	47	23 (29,1)	3	0	79	79
GCS	(36,71)		(59,49)		(3,8)			
GF	55	50 (65,8)	22 (28,2)	26 (34,2)	1	0	78	76
Gr	(70,51)				(1,3)			
GFS	54 (67,5)	56 (70)	26 (32,5)	24 (30)	0	0	80	80
GP	52 (65)	71 (88,8)	28 (35)	9 (11,3)	0	0	80	80
GPS	66 (82,5)	69 (86,3)	14 (17,5)	11 (13,8)	0	0	80	80
GS	22 (27,5)	46 (57,5)	57	34 (42,5)	1 (1,25)	0	80	80
GS			(71,25)					
GSS	21 (26,6)	46 (57,5)	55 (69,2)	34 (42,5)	3 (0	79	80
G33					3,8)			
TOTAL	328	433	295	202	13	0	636	635
	(51,6)	(68,2)	(46,4)	(31,8)	(2,04)			

Abbreviation: Thermo: thermocycling and storage before test.

3 CONCLUSÃO

Embora o ensaio de *push-out* seja naturalmente utilizado para avaliar resistência de união de retentores ao canal radicular de dentes despolpados, essa metodologia não permite que seja feita uma avaliação criteriosa de cada interface envolvida no complexo conjunto formado entre retentor, cimento e dentina radicular. O padrão de falhas existente nesse teste dificilmente será homogêneo entre diferentes grupos de teste. No caso de pinos anatômicos, a presença de mais um material nesse conjunto (resina composta) dificulta ainda mais a obtenção de resultados precisos. Levando isso em consideração, foi necessário que uma abordagem mais específica fosse feita para avaliar a resistência de união entre a resina composta do pino reembasado e o cimento resinoso autoadesivo.

A partir do teste de microtração, foi possível isolar a interface de interesse do estudo eliminado falhas entre o cimento resinoso e a dentina radicular e entre resina composta e os pinos de fibra de vidro. Com isso, obteve-se resultado preciso quanto a resistência de união entre resina composta e cimento resinoso autoadesivo. A partir dos nossos achados, foi possível concluir que o aumento da rugosidade de superfície da resina composta com o jateamento com óxido de alumínio melhorou a resistência de união com o cimento resinoso autoadesivo tanto nos testes imediatos quanto após a ciclagem térmica. No entanto, a associação da aplicação de silano a este jateamento mostrou melhores resultados apenas no teste imediato, não havendo diferença após a termociclagem. Por outro lado, na impossibilidade de realizar o tratamento mecânico sugerido, a utilização do agente silano é indicada.

Tanto o uso de peróxido de hidrogênio à 35% quando o ácido fluorídrico à 10% não geraram resultados melhores comparados ao grupo controle. Neste caso, parece desnecessário lançar mão de qualquer um desses métodos no tratamento de pinos anatômicos a serem cimentados com cimento resinoso autoadesivo.

Como limitação do estudo, apontamos a impossibilidade de obter resultados precisos no ensaio de *push-out* em virtude de as falhas terem sido distribuídas heterogeneamente entre várias interfaces presentes. No entanto, pode-se afirmar que, utilizando como base os resultados obtidos no teste de microtração, é possível ter maior segurança na aplicação da técnica de pinos anatômicos em situações clínicas. O jateamento da superfície dos pinos reembasados parece ser suficiente para que o cirurgião-dentista tenha segurança quanto à cimentação desses pinos. No entanto, a adesão do cimento resinoso autoadesivo à dentina radicular parece ser o elo mais fraco e, portanto, merece ser estudada.

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ANEXO A – NORMAS PARA A PUBLICAÇÃO NO PERIÓDICO *OPERATIVE* DENTISTRY

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EXAMPLES OF REFERENCE STYLE

- Journal article: two authors
 - Evans DB & Neme AM (1999) Shear bond strength of composite resin and amalgam adhesive systems to dentin *American Journal of Dentistry* **12(1)** 19-25.
- Journal article: multiple authors Eick JD, Gwinnett AJ, Pashley DH & Robinson SJ (1997) Current concepts on adhesion to dentin *Critical Review of Oral and Biological Medicine* **8(3)** 306-335.
- Journal article: special issue/supplement
 Van Meerbeek B, Vargas M, Inoue S, Yoshida Y, Peumans M, Lambrechts P & Vanherle G (2001) Adhesives and cements to promote preservation dentistry
 Operative Dentistry (Supplement 6) 119-144.

• Abstract:

Yoshida Y, Van Meerbeek B, Okazaki M, Shintani H & Suzuki K (2003) Comparative study on adhesive performance of functional monomers *Journal of Dental Research* **82(Special Issue B)** Abstract #0051 p B-19.

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