PRÓ-REITORIA DE PESQUISA E PÓS-GRADUAÇÃO
MESTRADO EM ODONTOLOGIA

GRACE MITIKO ROSATI HORI SATO

EFEITO DO PRÉ-TRATAMENTO DA DENTINA COM DIMETILSULFÓXIDO E DA TEMPERATURA DE VOLATILIZAÇÃO DO ADESIVO NA RESISTÊNCIA DE UNIÃO DE PINOS DE FIBRA

Presidente Prudente - SP
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Dissertação apresentada à Pró-Reitoria de Pesquisa e Pós-Graduação, Universidade do Oeste Paulista, como parte dos requisitos para obtenção do título de Mestre em Odontologia - Área de Concentração: Clínica Odontológica.

Orientador:
Prof. Dr. Anderson Catelan

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DEDICATÓRIA

Dedico este trabalho aos amores da minha vida, meus filhos Gabriel Yudi e Leonardo Eiji, companheiros de todas as horas, que me apoiaram incondicionalmente para que esta dissertação pudesse ser concluída. Que tiveram compreensão nos momentos que estive ausente. Amo vocês meus filhos!

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RESUMO

Efeito do pré-tratamento da dentina com dimetilsulfóxido e da temperatura de volatilização do adesivo na resistência de união de pinos de fibra

O pré-tratamento da dentina com dimetilsulfóxido (DMSO) pode resultar em maior infiltração resinosa e o ar aquecido para volatilizar o adesivo pode aumentar a evaporação dos solventes, melhorando a adesão à dentina radicular. O objetivo neste estudo “ex vivo” foi avaliar o efeito do pré-tratamento da dentina do conduto radicular com DMSO e da temperatura de volatilização de solventes de um sistema adesivo universal na resistência de união de pinos de fibra de vidro fixados com um cimento resinoso dual em dentes unirradiculares. Quarenta raízes bovinas (n = 10) com comprimento de 15 mm foram usadas, o canal radicular foi tratado endodonticamente e preparado (10 mm) para fixação de um pino de fibra de vidro (Exacto, Angelus) com o sistema de cimentação Single Bond Universal + RelyX Ultimate (3M ESPE). Inicialmente foi realizado o pré-tratamento da dentina com DMSO ou água destilada (controle) por 60 s e removido o excesso de umidade. Em seguida, o sistema adesivo foi aplicado no modo autocondicionante e realizada sua volatilização à 23ºC (temperatura ambiente) ou 40ºC (jato de ar aquecido) por 10 s. A fotoativação dos materiais foi realizada pelo tempo recomendado pelo fabricante com um LED *polywave* com irradiância de 1.000 mW/cm^2. Finalizada a cimentação dos pinos de fibra, os espécimes foram submetidos a ciclagem térmica (10.000 ciclos - 5 e 55ºC). Então, as raízes foram seccionadas para obtenção de fatias com 1 mm de espessura dos terços cervical, médio e apical. A resistência de união dos terços foi mensurada pelo ensaio de *push out* usando uma máquina de ensaio universal. O padrão de falha foi avaliado em estereomicroscópio. Os dados foram submetidos à ANOVA três critérios em parcelas subdivididas e teste de Tukey (α = 0,05). A volatilização do adesivo à 40ºC promoveu aumento na resistência de união comparado à temperatura de 23ºC. O terço cervical apresentou a maior resistência de união, seguido pelo terço médio, sendo a menor resistência de união observada no terço apical, com diferença estatística entre si. Desta forma, pode-se concluir que a volatilização de solventes do sistema adesivo usando jato de ar aquecido promove aumento da resistência de união do pino de fibra de vidro.

Palavras-chave: Adesão dentária, Adesivos, Cimentos resinosos.

ABSTRACT

Effect of dentin pretreatment with dimethyl sulfoxide and adhesive volatilization temperature on the bond strength of fiber posts

Dentin pretreatment using dimethyl sulfoxide (DMSO) can result in higher resin infiltration and warm air to volatilize the adhesive can increase solvent evaporation, improving adhesion to root dentin. The aim in this "ex vivo" study was to evaluate the effect of pretreatment of root canal dentin using DMSO and the volatilization temperature of solvents of a universal adhesive system on the bond strength of fiberglass posts fixed with a dual curing resin cement in single-rooted teeth. Forty bovine roots (n = 10) with a length of 15 mm were used, which the root canal was endodontically treated and prepared (10 mm) for fixation of a fiberglass post (Exacto, Angelus) with the luting system Single Bond Universal + RelyX Ultimate (3M ESPE). Initially, the dentin was pre-treated with DMSO or distilled water (control) for 60 s and excess moisture was removed. Then, the adhesive system was applied in the self-etching mode and its volatilization is carried out at 23ºC (room temperature) or 40ºC (warm air stream) for 10 s. Light curing of the materials was carried out for the time recommended by the manufacturer with a polywave LED at irradiance of 1,000 mW/cm^2. After the cementation of the fiber posts, the specimens were subjected to thermal cycling (10,000 cycles - 5 and 55ºC). Then, the roots were sectioned to obtain 1 mm thick slices of the cervical, middle, and apical thirds. Bond strength of the thirds was measured by the push out test using a universal testing machine. Failure pattern was evaluated under a stereomicroscope. Data were submitted to three-way split-plot ANOVA and Tukey's test (α = 0.05). Adhesive volatilization at 40ºC promoted an increase on bond strength compared to a temperature of 23ºC. The cervical third had the highest bond strength, followed by the middle third, with the lowest bond strength observed in the apical third, with statistical difference between them. Thus, it can be concluded that the solvent volatilization of adhesive system using warm air stream promotes an increase on bond strength of fiberglass post.

Keywords: Dental adhesion, Adhesives, Resin cements.

LISTA DE SIGLAS E SIMBOLOS

DMSO	-	Dimetilsulfóxido
LED	-	*Light Emitting-diode*
H_2O-23	-	Grupo experimental com pré-tratamento água e temperatura de volatilização de 23ºC
H_2O-40	-	Grupo experimental com pré-tratamento água e temperatura de volatilização de 40ºC
DMSO-23	-	Grupo experimental com pré-tratamento de dimetilsulfóxido e temperatura de volatilização de 23ºC
DMSO-40	-	Grupo experimental com pré-tratamento de dimetilsulfóxido e temperatura de volatilização de 40ºC
ANOVA	-	Análise de variância
º	-	Graus
ºC	-	Graus Celsius
=	-	Igual a
%	-	Porcentagem
ª	-	Segunda
mL	-	Mililitros
nº	-	Número
h	-	Hora
mm	-	Milímetros
mW/cm2	-	Miliwatt por centímetro quadrado
#	-	Número
s	-	Segundos
:		Proporção
KgF	-	Quilograma-força
mm/s	-	Milímetros por segundo
MPa	-	Mega Pascal
x		Multiplicação
ρ	-	Rô
≤	-	Menor ou igual
>	-	Maior que
<	-	Menor que
Corp.	-	*Corporation*

LISTA DE FIGURAS

LISTA DE TABELA

SUMÁRIO

ARTIGO CIENTÍFICO

Effect of dentin pretreatment using DMSO and adhesive volatilization temperature on bond strength of fiber posts

Abstract

The aim in this "ex vivo" study was to evaluate the effect of pretreatment of root canal dentin using DMSO and the volatilization temperature of solvents of a universal adhesive system on the bond strength of fiberglass posts fixed with a dual cure resin cement in single-rooted teeth. Forty bovine roots (n = 10) with a length of 15 mm were used, which the root canal was endodontically treated and prepared (10 mm) for fixation of a fiberglass post (Exacto, Angelus) with the luting system Single Bond Universal + RelyX Ultimate (3M ESPE). Initially, the dentin was pre-treated with DMSO or distilled water (control) for 60 s and excess moisture was removed. Then, the adhesive system was applied in the self-etching mode and its volatilization is carried out at 23ºC (room temperature) or 40ºC (warm air stream) for 10 s. Light curing of the materials was carried out for the time recommended by the manufacturer with a polywave LED at irradiance of 1,000 mW/cm^2. After the cementation of the fiber posts, the specimens were subjected to thermal cycling (10,000 cycles - 5 and 55ºC). Then, the roots were sectioned to obtain 1 mm thick slices of the cervical, middle, and apical thirds. Bond strength of the thirds was measured by the push out test using a universal testing machine. Failure pattern was evaluated under a stereomicroscope. Data were submitted to three-way split-plot ANOVA and Tukey's test ($\alpha = 0.05$). Adhesive volatilization at 40ºC promoted an increase on bond strength compared to a temperature of 23ºC. The cervical third had the highest bond strength, followed by the middle third, with the lowest bond strength observed in the apical third, with statistical difference between them. Thus, it can be concluded that the solvent volatilization of adhesive system using warm air stream promotes an increase on bond strength of fiberglass post.

Keywords: Dental adhesion, Adhesives, Resin cements.

Introduction

After completion of endodontic treatment, it is commonly necessary to use an intraradicular retainer to provide adequate support and retention of restoration in cases with significant coronary destruction.[1,2] Fiberglass post (FGP) is esthetic, it has bonding to resin cement, allowing cementation in a single session and soon after endodontic treatment is completed, elastic modulus similar to dentin, with better stress distribution and less chance of fracture.[1,3]

Despite the advantages of FGP, it can present adhesion failures inside the root canal, which can lead to its displacement.[2,3] Relining the FGP with resin composite provides better retention, due to better adaptation and decrease of the thickness of resin cement, especially in flared canals.[1,3] In addition, the FGP cementation technique should be taken into account to increase the success rate of this procedure. Dual resin cements are the materials of choice for cementing FGPs, having favorable characteristics of physical polymerization by light and chemical polymerization in its absence, ensuring adequate mechanical properties.[4,5]

Bond strength reduction has been attributed to combined degradation of exposed collagen and resin polymer at adhesive interface.[6,7] Incomplete infiltration of monomers into demineralized dentin zone, both in etch-and-rinse and self-etch adhesive systems has been reported, being observed the permanence of collagen fibers exposed and, therefore, not impregnated by fluid resin.[7-9]

One of methods to reduce this failure is the use of a warm air stream for solvent evaporation of adhesive system in order to improve the bond strength.[10] Since warm air increases the evaporation rate of solvents and residual water, in addition to increasing the kinetic energy of molecules, improving the mechanical properties of adhesive and the quality of hybrid layer, with a more stable bond to dentin in long term.[11-14]

Another approach to improve the adhesion of resin-based materials is the use of dimethyl sulfoxide (DMSO).[15-19] This solvent is not very toxic and capable of dissolving polar and nonpolar substances, such as resin monomers.[16,20] DMSO is considered a of best tissue penetration enhancers, as it can replace water molecules, increasing interfibrillar spaces, providing increased resin infiltration and higher stability of resin-dentin interface.[15,17,20-26]

Thus, it is extremely important that the dentist has knowledge about effective protocols to complete the rehabilitation procedure of endodontically treated teeth, especially when there is a great loss of tooth structure. Therefore, the combination of the use of a warm air to more effectively volatilize the solvent of adhesive system, improving its physical properties and dentin pretreatment using DMSO to increase resin infiltration, could reduce the degradation of resin-dentin bonding interface and increase the longevity of adhesive restorative procedures.

The aim in this study was to evaluate the effect of dentin pretreatment of root canal using DMSO and solvent volatilization temperature of a universal adhesive system on the bond strength of fiberglass posts fixed using a conventional dual resin cement in single root canals, as well as their failure pattern, in each root third. The research hypotheses were that the (1) dentin pretreatment using DMSO and (2) use of warm air stream to solvent volatilization of adhesive would increase the bond strength.

Material and method

Experimental design

The experimental design in this study consisted of two factors: dentin pretreatment (two levels: distilled water and DMSO) and temperature of solvent volatilization of adhesive (two levels: 23ºC and 40ºC); and one subfactor: root third (three levels: cervical, middle, and apical). The response variable was bond strength.

Teeth selection

In this study, 40 bovine roots (n = 10), single-rooted, straight, with similar dimensions, recently extracted, closed apices, without cracks, and without shape changes were used. Teeth were disinfected in 0.1% thymol for 7 days and stored in distilled water at 37°C for up to 3 months. After this step, the roots were marked, standardized at a length of 15 mm, and sectioned using a diamond-cutting disc (Buehler, Lake Bluff, IL, USA) that was attached to a high-speed precision saw (Biopdi, São Carlos, SP, Brazil), in order to separate the roots from their crowns.

Endodontic treatment

The roots were instrumented to working length, 1 mm short of apex, using K-files (Dentsply/Maillefer, Ballaigues, Switzerland), and the teeth were instrumented by serial technique using 1st series (#15 - #40) and 2nd series (#45-#80) in working length. At each instrument change, the root canals were irrigated with 5 mL of physiological saline solution. After instrumentation, the root canals were dried with #80 absorbent paper cones. Obturation was performed using the active lateral condensation technique, using gutta-percha cones (R3, R5, and accessory cones/Dentsply, Petrópolis, RJ, Brazil) and AH Plus endodontic cement (Dentsply). After completion of endodontic treatment, the canals were sealed with a zinc oxide temporary filling material without eugenol (Coltosol; Vigodent, Rio de Janeiro, RJ, Brazil). Endodontically treated roots were stored in distilled water for 7 days at 37ºC.

Intraradicular preparation

Intraradicular preparation was performed using a Gates-Glidden bur #2 to 5 (Dentsply/Maillefer) for removal of 10 mm long gutta-percha, controlled by a cursor, corresponding to the length of FGP inside the root, remaining 4 mm of filling in the root canal. Then, the sequence of drills (#1 - 3) from Exacto FGP kit (Angelus, Londrina, PR, Brazil) was inserted in a single movement, inside the canal up to a length of 10 mm, cleaned with distilled water and dried. Then, a standard post was positioned inside it to verify the correct preparation of the root canal. After completion of intraradicular preparation, irrigation with distilled water (5 mL) was performed to remove the dentinal "debris", drying with absorbent paper cones nº 80 (Dentsply).

Surface treatment and relining of FGP with resin composite

For all experimental groups, FGP Exacto nº 3 (Angelus) were previously cleaned with sterile gauze moistened with 70% ethanol and Prosil silane (FGM Dental Group, Joinville, SC, Brazil) was applied for 1 min according to manufacturer's recommendation. Then, solent free Scotchbond Multipurpose adhesive (3M ESPE, St. Paul, MN, USA) was applied and light-cured for 20 s using a third-generation LED (light emitting-diode) device (Valo; Ultradent Inc., South Jordan, UT, USA), at an irradiance of 1,000 mW/cm^2, monitored by a radiometer (model L.E.D. Radiometer; Kerr Corporation, Middleton, WI, USA). Resin composite (shade A1; Filtek One Bulk Fill, 3M ESPE) was then placed around the FGP and the set was placed in the canal lubricated with water-soluble gel (K-Y, Johnson & Johnson, São José dos Campos, SP, Brazil), and the light was cured for 20 s, the relined FGP was removed, the light curing was completed for another 20 s.[1]

Luting procedure

For groups in which dentin was pre-treated with DMSO, 50% DMSO solution was applied actively for 60 s[16] and the excess removed with absorbent paper cones.

For control group, distilled water was applied as described above. Subsequently, a layer of Single Bond Universal Adhesive System (3M ESPE) was actively applied for 20 s, using a micro-applicator. Then, excess adhesive was removed with absorbent paper points and the solvents/residual water were volatilized using an air stream for 10 s at two different temperatures: 23ºC (room temperature) or 40ºC (warm air) using a portable device (Erios, São Paulo, SP, Brazil).

Resin cement RelyX Ultimate (3M ESPE) was dispensed in a proportion of 1:1 on a block of waterproof paper and mixed until the luting agent was completely homogenized. Then, it was inserted into the root canal using needle tip of “Centrix” syringe. Then, the FGP was positioned, manually stabilized inside the root canal and the excess cement was removed using a micro-applicator. After 5 min, light curing was performed using Valo (Ultradent Inc.) curing unit, 40 s on buccal surface and 40 s per lingual/palatal surface.

Thermal cycling

After the luting procedure, specimens were submitted to 10,000 thermal cycles (OMC300 TSX; Odeme Dental Research, Luzerna, SC, Brazil) in alternating baths at 5 and 55ºC for 30 s of permanence at each temperature and 15 s of transfer time.[27]

Sectioning the roots into thirds

To perform the cuts, the roots were fixed with compound in acrylic plates and then sectioned perpendicularly to their long axis with a diamond-cutting disc (Buehler) that was attached to a high-speed precision saw (Biopdi), in order to obtain two slices with a thickness of approximately 1.1 mm from each root third (cervical, medium, and apical), totaling six slices of each specimen.

For the push out bond strength test, the slices were identified with a pencil on the cervical face and kept in distilled water in a microtube at 37°C for 24 h, according to the respective group and root third.

Bond strength

Specimens were placed on a stainless-steel metallic base containing a 3 mm internal diameter hole in the central region. Root sections containing the FGP were positioned exactly in the same direction as the hole in metal base. The entire set was positioned on base of a universal testing machine (23-2S; INSTRON-EMIC, São José dos Pinhais, PR, Brazil) equipped with a load of 50 KgF. A metal rod with a 1 mm diameter live tip fixed to the machine jaw and positioned in center of FGP. Push out test was conducted at a speed of 1 mm/s until fracture. Force required for fracture was obtained in N and converted into MPa by dividing the force by adhesive area of root canal. Adhesive area = (R1 + R2) $[(R1 + R2)2 + h2]^{1/2}$; where R1 represents the coronal root canal radius, R2 represents the apical root canal radius, and h represents the slice thickness.[28]

Fracture pattern analysis

After push out testing, root slices were analyzed using a stereoscopic microscope (SZB-STMPRO-B; Bel Photonics, Piracicaba, SP, Brazil) at 40x magnification to observe the failure mode. Fracture pattern were classified into: (1) adhesive failure between root dentin and resin cement, (2) adhesive failure between resin cement/relined FGP, (3) cohesive failure in root dentin, (4) cohesive failure in resin cement/FGP/relining resin composite, and (5) mixed failure (involving more than one pattern previously described).[29,30]

Statistical analysis

Data normality and homogeneity were analyzed using the Kolmogorov-Smirnov and Levene tests, respectively. Then, data were submitted to three-way analysis of variance (ANOVA) in split plot for root thirds, followed by Tukey's test for multiple comparisons. In all analyses, significance level was established at 5% (Assistat 7.7, Campina Grande, PB, Brazil).

Results

ANOVA showed a statistical difference for factors volatilization temperature ($\rho = 0.0101$) and root thirds ($\rho < 0.0001$). For the factor dentin pretreatment using DMSO ($\rho = 0.7324$), as well as for all the interactions of the factors, no significant difference was observed ($\rho > 0.05$). Adhesive volatilization at 40ºC showed an increase on bond strength compared to temperature of 23ºC. In the comparison between the root thirds, the cervical third presented the highest bond strength, followed by the middle third, with the lowest bond strength observed in the apical third, with statistical difference between them (Table 1).

Table 1 - Bond strength (MPa) means (standard deviation) according to dentin pretreatment using dimethyl sulfoxide, adhesive volatilization temperature, and root thirds.

Volatilization temperature	Dentin pretreatment	Root third			Pooled data
		Cervical	Middle	Apical	
23ºC	Distilled water	14.56 (3.58)	13.16 (3.59)	6.73 (2.16)	12.24 (4.58) b
	DMSO	15.16 (3.30)	14.02 (4.70)	9.78 (3.71)	
40ºC	Distilled water	18.44 (4.48)	15.42 (3.24)	11.15 (2.89)	14.56 (4.54) a
	DMSO	17.03 (4.94)	14.38 (3.61)	10.96 (2.71)	
Pooled data		16.30 (4.26) A	14.24 (3.77) B	9.66 (3.33) C	

Different letters (uppercase comparing the root thirds and lowercase letters comparing the volatilization temperatures) indicate a statistical difference ($\rho \leq 0.05$).

Fracture pattern in percentage is shown in Figure 1. The most frequent failure mode was the adhesive between root dentin and resin cement, followed by mixed failure, cohesive in dentin, and adhesive between resin cement and relined FGP.

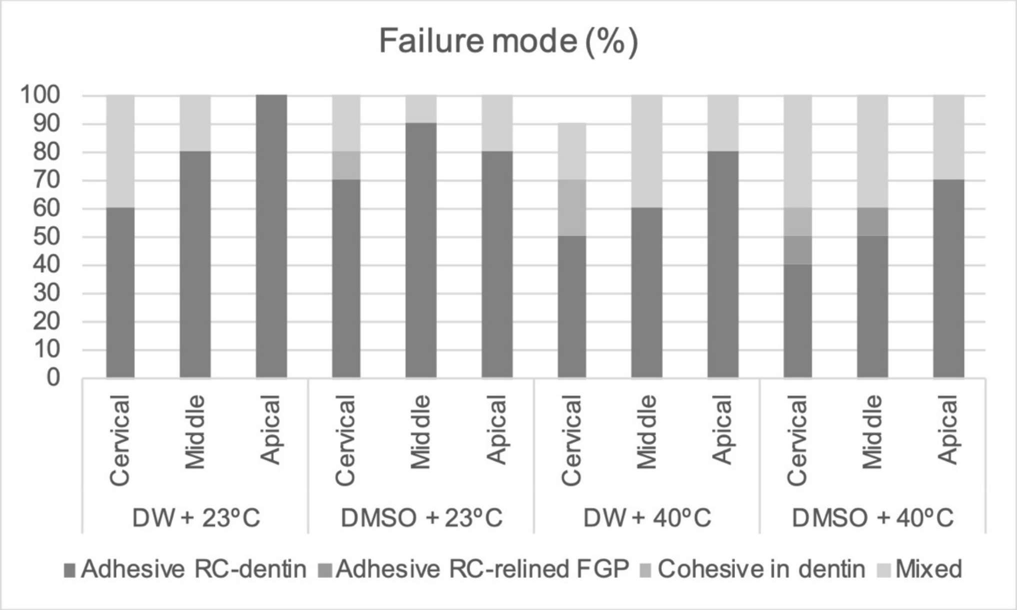

Figure 1 - Failure mode (%) according to dentin pretreatment using dimethyl sulfoxide, adhesive volatilization temperature, and root thirds. *DW: distilled water; DMSO: dimethyl sulfoxide; RC: resin cement; FGP: fiberglass post.

Discussion

The quality of the adhesive interface between the dentin tissue and the luting agent is one of the main factors responsible for success of rehabilitative treatment using GFP.[31,32] An optimized infiltration of monomers into demineralized collagen zone in dentin favors the formation of a highly cross-linked polymer.[13] However, for this process to occur without prejudice, the effective removal of residual dentin water and solvent from the adhesive system are essential previously the light curing.[14,33]

The failure commonly found is related to displacement of FGP inside the canal, this is mainly due to the degradation of resin-dentin interface and hydrolysis of the resin-based material when functioning in oral cavity. The combinations of the humid environment, with thermal variations and exposed to masticatory functions are part of the clinical scenario that are simulated by *in vitro* studies to simulate the aging of this interface.[34] Matrix metalloproteinases (MMPs) act degrading the collagen matrix of hybrid layer,[2,17] moreover the anatomical characteristics of a narrow, deep root space with different density of dentinal tubules may also contribute to this degradation.[35,36]

DMSO is a polar aprotic solvent that acts by increasing the penetration capacity of monomers between fibrillar spaces by altering the behavior of water. This ability is achieved by a two-hydrogen bond with water, which prevents auto-associative activity and promotes the reduction of residual water trapped between the polymer chains. Improvement in collagen wetting by monomers represents greater hybridization and consequently improves the bond strength.[17]

In this study, dentin pretreatment of root canal with DMSO showed no improvement on bond strength, so the first hypothesis was rejected. Previous studies also did not observe an increase on bond strength of adhesive restorations when a self-etch adhesive[37] or etch-and-rinse[17] were used, corroborating with the present investigation that used a conventional dual curing luting system that uses a self-etch adhesive. However, DMSO reduced the zone of exposed collagen[37] and preserved the stability of the hybrid layer, in addition significantly inhibited the MMP activity.[17] Another study, also observed no difference on immediate bond strength when acid-etched root dentin was treated with DMSO, but this procedure preserved the stability of adhesion over time when GFP was luted with etch-and-rinse adhesive resin cements.[2]

Currently, adhesive systems have solvents in their composition that have the function of facilitating the diffusion of monomers into collagen matrix, such as water and/or ethanol or acetone.[13] However, there is a need to eliminate these solvents before light curing of adhesive system, as their presence can affect the strength of the hybrid layer.[13]

In clinical routine, solvent evaporation is usually performed using a warm air stream at room temperature, but as a way of accelerating this process, volatilization with heated air has been a strategy for more effective evaporation of solvents and residual water of dentin with improvement on physical properties and bond strength.[13,14,33] In this study, solvent volatilization using a air-stream heated at 40ºC promoted an increase on bond strength when compared to volatilization at 23ºC, so the second hypothesis was accepted.

Comparing the root thirds, there was a gradual reduction in bond strength values, with the cervical third showing the highest adhesion value and the apical third the lowest value. The different densities of dentinal tubules present along the root canal, in addition to the anatomical conditions that do not favor the perfect execution of the technique as the depth increases, the presence of possible residues of filling material in this portion.[36] Another important issue is the polymerization quality, since degree of conversion is reduced by attenuation and scattering of curing light at the deeper root-canal level, and consequently lower mechanical properties of resin cement are obtained.[36]

The most frequent failure mode was the adhesive between root dentin and resin cement, followed by mixed failure, cohesive in dentin, and adhesive between resin cement and relined FGP. Warm air volatilization provided a reduction in adhesive failures, possibly due to an increase in bond strength values, by increased kinetic energy of molecules and consequently improved mechanical properties of adhesive system.[11-14] It was not observed cohesive failure in resin cement/FGP/relining resin composite and there was no failure in adhesion between post and relining resin, evidencing that the weakest link of FGPs is still the root dentin-resin cement bonding.

Although bovine teeth are considered adequate substitutes for human teeth, it represents a limitation. In addition, it is known that the results of laboratory studies do not always reflect what would occur in a clinical situation. However, the results obtained in this investigation may encourage future studies to evaluate the combination of dentin pretreatment with DMSO and warm air volatilization of adhesive with longer aging periods and clinical studies.

Conclusion

The volatilization of solvents of adhesive system using warm air stream promoted an increase on bond strength of fiberglass post, which could increase the clinical longevity of this procedure.

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APÊNDICE - METODOLOGIA ILUSTRADA

DELINEAMENTO EXPERIMENTAL

Unidades experimentais: raízes unirradiculares bovinas nas quais foi realizada a cimentação de pinos de fibra de vidro.

Fatores em estudo:

Pré-tratamento da dentina com DMSO em dois níveis:

- Sim;
- Não.

Temperatura de volatilização do adesivo em dois níveis:

- 23ºC (temperatura ambiente);
- 40ºC.

Subfator em estudo:

Terço da raiz em três níveis

- Cervical;
- Médio;
- Apical.

Variável de resposta: resistência de união por *push out*.

Forma de designação: processo aleatório por meio de sorteio.

CONFECÇÃO DOS ESPÉCIMES

Seleção dos dentes e padronização dos espécimes

Neste estudo foram utilizadas 40 raízes bovinas (n = 10), unirradiculares, retas, com dimensões similares, recém-extraídas, ápices fechados, sem trincas e sem alterações de forma. Os dentes foram desinfetados em timol 0,1% por 7 dias e armazenados em água destilada a 37ºC por até 3 meses. Após esta etapa, as raízes foram marcadas e padronizadas no comprimento de 15 mm e seccionadas com disco diamantado em cortadeira metalográfica, com o objetivo de separar as raízes de suas coroas (Figuras 2A-2E).

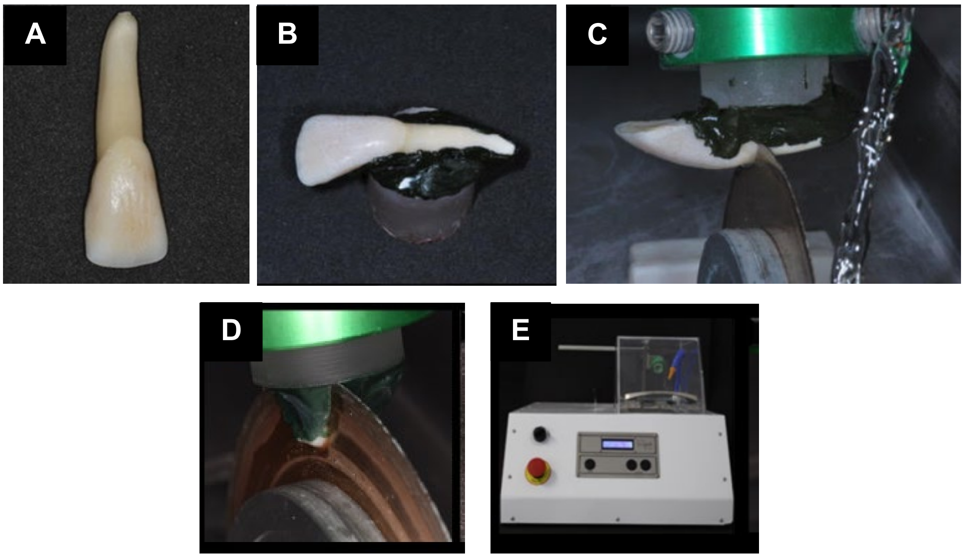

Figura 2 - A) Dente bovino; B) Dente bovino fixado com godiva de baixa fusão; C) Posicionamento do dente e fixação no dispositivo para corte; D) Corte sendo realizado; E) Cortadeira metalográfica (Fonte: arquivo pessoal)

<u>Técnica endodôntica</u>

As raízes foram instrumentadas até o comprimento de trabalho, 1 mm aquém do ápice, com limas do tipo K (Dentsply/Maillefer, Ballaigues, Suíça), sendo os dentes instrumentados pela técnica seriada utilizando 1ª série (#15 - #40) e 2ª série (#45- #80) no comprimento de trabalho (Figura 3). A cada troca de instrumento, os canais radiculares foram irrigados com 5 mL de solução salina fisiológica. Após a instrumentação, os canais radiculares foram secos com cones de papel absorventes nº 80.

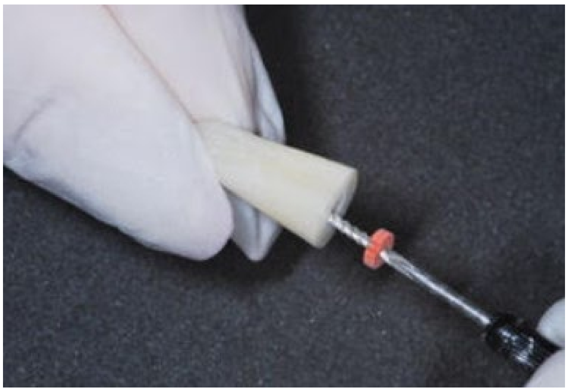

Figura 3 - Instrumentação das raízes ate o comprimento de trabalho. (Fonte: arquivo pessoal).

A obturação foi realizada pela técnica de condensação lateral ativa (Figura 4), utilizando-se cones de guta-percha (R3, R5 e cones acessórios/Dentsply, Petrópolis, RJ, Brasil) e o cimento endodôntico AH Plus (Dentsply).

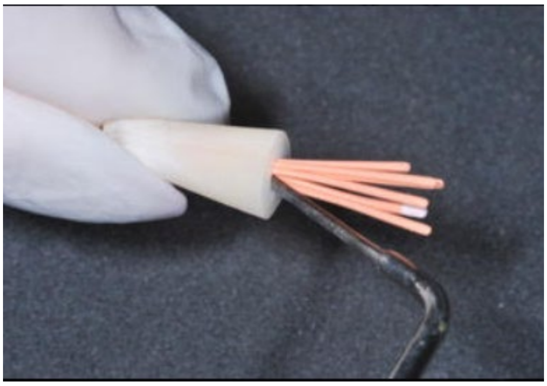

Figura 4 - Obturação pela técnica da condensação lateral ativa. (Fonte: arquivo pessoal)

Após a finalização do tratamento endodôntico os canais foram vedados com selador a base de óxido de zinco sem eugenol (Coltosol; Vigodent, Rio de Janeiro, RJ, Brasil) (Figura 5). As raízes foram armazenadas em água destilada por 7 dias em estufa a 37ºC.

Figura 5 - Material selador a base de óxido de zinco sem eugenol. (Fonte: www.dentalcremer.com.br)

Preparo intrarradicular

O preparo intrarradicular foi realizado com broca Gates-Glidden nº 2 a 5 (Dentsply/Maillefer) para a remoção de guta-percha no comprimento de 10 mm, controlado por cursor, correspondendo ao comprimento do pino no interior da raiz, permanecendo 4 mm de obturação no canal radicular. Em seguida a sequência de brocas (#1 - 3) do próprio kit do pino Exacto (Angelus, Londrina, PR, Brasil) foi inserida em movimento único (Figuras 6A-6B), no interior do canal até o comprimento de 10 mm, feita a limpeza com água destilada e secagem. Em seguida, um pino padrão foi posicionado no interior do mesmo para se verificar o correto preparo do conduto radicular. Após a finalização do preparo intrarradicular, realizamos a irrigação com água destilada (5 mL) para remoção dos “debris” dentinários, secagem com cones de papel absorventes nº 80 (Dentsply).

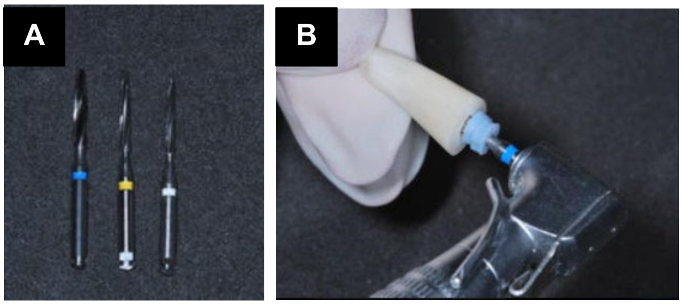

Figura 6 - A) Broca do sistema de pinos; B) Broca finalizando o preparo do conduto. (Fonte: arquivo pessoal)

Tratamento de superfície e reembasamento com resina composta do pino de fibra de vidro

Para todos os grupos experimentais os pinos de fibra de vidro Exacto nº 3 (Angelus) previamente limpos com gaze estéril umedecida com álcool 70% e realizada a aplicação do silano (Angelus) por 1 min conforme as recomendações do fabricante do pino (Figura 7A). Em seguida, foi aplicado o adesivo puro do Scotchbond Multipurpose (3M ESPE, St. Paul, MN, EUA) (Figura 7B), o qual fotoativado por 20 s usando um aparelho LED (*light emitting-diode*) de terceira geração (Valo; Ultradent Inc., South Jordan, UT, EUA), com irradiância de 1.000 mW/cm^2, com monitoramento constante por meio de radiômetro (modelo L.E.D. Radiometer; Kerr Corp., Middleton, WI, EUA).

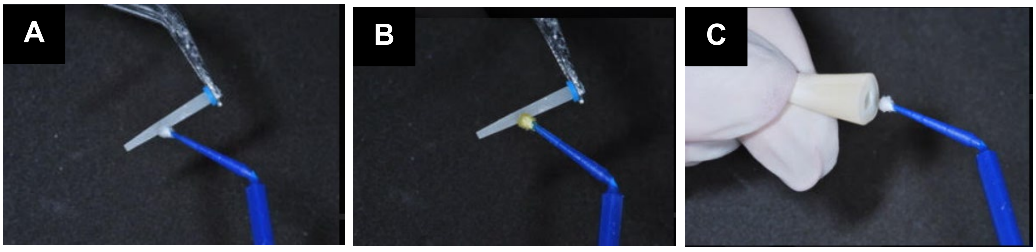

Figura 7 - A) Aplicação do silano no pino de fibra de vidro; B) Aplicação do adesivo; C) Lubrificação do conduto radicular com gel hidrossolúvel (Fonte: arquivo pessoal)

Então foi colocada a resina composta (Filtek One Bulk Fill, 3M ESPE) ao redor do pino e o conjunto foi levado ao conduto lubrificado com gel hidrossolúvel (K-

Y, Johnson & Johnson, São José dos Campos, SP, Brasil) (Figura 7C), realizada a fotoativação por 20 s, removido o pino reembasado foi complementada a fotoativação por mais 20 s (Figuras 8A-8C).

Figura 8 - A) Modelagem do pino de fibra de vidro com resina composta; B) Fotoativação no conduto radicular; C) Complementação da fotoativação fora do conduto radicular. (Fonte: arquivo pessoal)

<u>Procedimentos adesivo</u>

Para os grupos em que a dentina foi pré-tratada com DMSO (Figura 9), foi aplicado ativamente a solução de 50% de DMSO por 60 s e removido o excesso com cones de papel absorvente nº 80.

Figura 9 - Solução de DMSO usada. (Fonte: arquivo pessoal)

Para o grupo controle, realizamos aplicação de água destilada conforme descrito anteriormente. Posteriormente aplicamos uma camada do sistema adesivo Single Bond Universal (3M ESPE) de forma ativa por 20 s, com auxílio de pincel do tipo *microbrush*. Então, o excesso de adesivo foi removido com pontas de papel absorventes #40 e a volatilização dos solventes realizada com um jato de ar por 10 s em duas diferentes temperaturas: 23ºC (temperatura ambiente) ou 40ºC (ar aquecido) usando um aparelho portátil (Erios, São Paulo, SP, Brasil) (Figura 10).

Figura 10 - Equipamento usado para volatilização do sistema adesivo nas temperaturas de 23 e 40ºC. (Fonte: arquivo pessoal)

O cimento resinoso RelyX Ultimate (3M ESPE) dispensado na proporção 1:1 sobre um bloco de papel impermeável e espatulado até a completa homogeneização do agente de cimentação. A seguir, o mesmo foi inserido no interior do conduto radicular com auxílio da ponta agulhada da seringa do tipo “Centrix”. Então, o pino de fibra de vidro foi posicionado (Figuras 11A-11C), estabilizado manualmente no interior do canal radicular e os excessos do cimento removidos com *microbrush*. Após 5 min, a fotoativação foi realizada com o aparelho Valo (Ultradent Inc.), 20 s na face vestibular e 20 s por lingual/palatina da raiz. A irradiância foi monitorada com um radiômetro (modelo L.E.D; Kerr Corp.).

Figura 11 - A) Cimento resinoso; B) Inserção do cimento no conduto radicular com a ponta agulhada da seringa tipo Centrix; C) Fixação do pino de fibra de vidro. (Fonte: arquivo pessoal)

CICLAGEM TÉRMICA

Após o procedimento de cimentação, os espécimes foram submetidos à 10.000 ciclos térmicos (OMC300 TSX; Odeme Dental Research, Luzerna, SC, Brasil) em banhos alternados de 5 e 55ºC durante 30 s de permanência em cada temperatura e 15 s de tempo de transferência (Figura 12).

Figura 12 - Máquina de termociclagem. (Fonte: arquivo pessoal)

Seccionamento das raízes em terços

Para a realização dos cortes, as raízes foram fixadas com godiva em placas de acrílico e posteriormente seccionadas perpendicularmente ao seu longo eixo com disco diamantado (Buehler, Lake Bluff, IL, EUA) que foi acoplado à cortadeira metalográfica (Biopdi, São Carlos, SP, Brasil), a fim de se obter duas fatias com espessura de aproximadamente 1,1 mm de cada terço da raiz: cervical, médio e apical, totalizando seis fatias de cada espécime (Figuras 13A-13C).

Figura 13 - A) Seccionamento das raízes em terços; B) Cortes realizados; C) Fatias obtidas. (Fonte: arquivo pessoal)

Para o teste de resistência de união por *push out*, as fatias foram identificadas com lápis na face voltada para cervical e mantidas em água destilada em microtubo em estufa a 37ºC por 24 h, conforme o respectivo grupo e terço.

Ensaio de resistência da união

Os espécimes foram posicionados em uma base metálica em aço inoxidável contendo um orifício de 3 mm de diâmetro interno na região central. As secções radiculares contendo o pino de fibra de vidro foram posicionadas exatamente na mesma direção do orifício da base metálica. Todo o conjunto foi posicionado na base da máquina de ensaio universal (23-2S; INSTRON-EMIC, São José dos

Pinhais, PR, Brasil) dotada de carga de 50 KgF. Uma haste metálica com ponta ativa de 1 mm de diâmetro fixada no mordente da máquina e posicionada no centro do pino de fibra de vidro (Figuras 14A-14B). O ensaio de *push out* foi conduzido à velocidade de 1 mm/s até a fratura. A força necessária para a fratura obtida em N e convertida em MPa dividindo-se a força pela área adesiva do canal radicular. Área adesiva = $(R1 + R2)\ [(R1 + R2)^2 + h^2]^{1/2}$; onde R1 representa o raio do canal radicular coronal, R2 representa o raio do canal radicular apical e h representa a espessura da fatia.

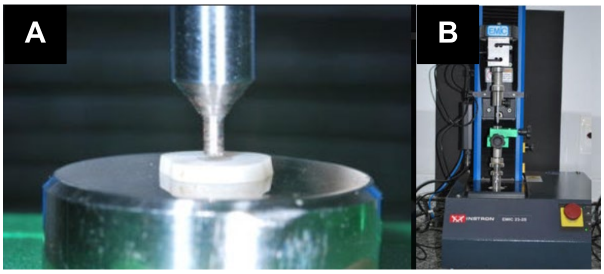

Figura 14 - A) Ensaio de *push out*; B) Máquina de ensaio universal. (Fonte: arquivo pessoal)

Análise do padrão de fratura

Após o ensaio mecânico de *push out*, os espécimes foram analisados utilizando um microscópio estereoscópio (SZB-STMPRO-B; Bel Photonics, Piracicaba, SP, Brasil) com aumento de 40x para observar o modo de falha (Figura 15). Sendo classificados em: (1) falha adesiva entre o cimento resinoso e a dentina radicular, (2) falha adesiva entre o cimento resinoso e o pino de fibra de vidro reembasado, (3) falha coesiva na dentina radicular, (4) falha coesiva no cimento resinoso/pino de fibra/resina composta do reembasamento, e (5) falha mista (envolvendo mais de um padrão anteriormente descrito).

Figura 15 - Microscópio estereoscópio. (Fonte: arquivo pessoal)

ANEXO - Normas do periódico “Journal of Prosthodontics”