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Push-out Bond Strength of Root-end Filling Materials

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The aim of this study was to evaluate the bond strength of root-end filling materials. Forty 2-mm-thick slices were obtained from human single-rooted teeth. After root canal preparation using a 1.5 mm diameter cylindrical drill, the dentinal walls were prepared by diamond ultrasonic tip (CVD T0F-2). The specimens were divided according the material (n=10): MTA Angelus (MTAA), MTA Sealer (MTAS, experimental), Sealer 26 (S26) and zinc oxide and eugenol cement (ZOE). The push-out test was performed in a mechanical test machine (EMIC DL 2000) at 1 mm/min speed. The failure type was evaluated by stereomicroscopy. The results were subjected to ANOVA and Tukey test, at 5% significance level. MTAA (19.18 MPa), MTAS (19.13 MPa) and S26 (15.91 MPa) showed higher bond strength ($p<0.05$). ZOE (9.50 MPa) showed the least bond strength values ($p<0.05$). Adhesive failure was prevalent in all groups, except for ZOE, which showed mixed failures. It was concluded that root-end filling materials MTA Angelus, MTA Sealer and Sealer 26 showed higher bond strength to dentinal walls than zinc oxide and eugenol cement after retrograde preparation.

Key Words: bond strength, root-end filling material, mineral trioxide aggregate, push-out.

Introduction

Push-out mechanical tests have been used to evaluate bond strength of filling materials and posts to root dentin (1). However, few studies evaluated the bond strength of root-end filling materials to dentinal walls after retrograde preparation. Bond strength of MTA and a new root-end filling material was evaluated in retrograde cavities prepared with either ultrasound or laser, and higher bond strength values were observed for cavities prepared by ultrasound (2).

The adaptation between retrograde filling material and dentin is important for the success of retrograde filling. The bond strength between root dentin and root-end filling material should provide adaptation and increased interface between material and dentin (1). Thus, the bond strength of endodontic materials has been evaluated by push-out tests. An ideal root-end filling material should have dimensional stability, radiopacity, proper setting time, antimicrobial activity, biocompatibility and ability to stimulate mineralized tissue (3). Several materials have been used as retro-end filling materials.

MTA-based root-end filling materials have been developed (4,5). An experimental sealer (MTA Sealer) was developed based on white Portland cement, zirconium oxide (radiopaque filler), calcium chloride (additive) and resin to be used as a root canal filling material (5). The addition of calcium chloride to MTA-based materials increases calcium releasing capacity (5). MTA Sealer has demonstrated calcium ions release (6), proper setting time, flow (7) and biocompatibility similar to MTA, after a study in rats (8).

Sealer 26 is also an epoxy resin-based endodontic sealer, composed by bismuth oxide, calcium hydroxide and epoxy resin. A greater powder/resin ratio was used to obtain consistency for retrograde fillings. Sealer 26 is known for its excellent sealing properties, when used either as root canal sealer or in root-end fillings (9). This material also has biocompatibility if used as root-end filling material (10), proper radiopacity and calcium ion release (11).

Conventional tips with a diamond-coated surface are widely used for root end-preparations. An improved type of diamond-coated tip is fabricated using the chemical vapor deposition method (CVD). The surface characteristic of the CVD tip may result in qualitative difference for root-end preparation (12,13)

This study evaluated the bond strength of root-end filling materials. The null hypothesis was that there are no differences in bond strength values among the materials in retrograde cavities prepared with ultrasonic tips.

Material and Methods

The composition and manufacturers of the evaluated root-end filling materials are in Table 1. MTA Angelus was mixed at 3:1 ratio in weight (powder-to-liquid), according to the manufacturer. MTA Sealer was mixed at 5:1 ratio in weight (1 g powder/ 0.2 mL resin) to obtain consistency of retrofilling material. Sealer 26 was mixed at 5:1 ratio in weight (1 g powder/0.2 mL resin), the consistency indicated for retrofilling (9). ZOE cement was mixed at 5:1 ratio (1 g powder/0.2 mL liquid) as indicated for retrofilling, according to Silva et al. (14).

Single-rooted human teeth were used. For selection, the teeth were previously radiographed (GE 1000, General Eletric, Milwaukee, WI, USA). They were maintained in 0.5% chloramine-T trihydrate solution (Fórmula & Ação Farmácia Magistral, São Paulo, SP, Brazil) for one week and then in distilled water at 4 °C, according to ISO/TS 11405:200 guidelines. The roots were embedded in polyester resin. Forty 2-mm sections were obtained. The root canal walls of each section were prepared by a 1.5 mm diameter cylindrical drill (Vortex Produtos Odontológicos, São Paulo, SP, Brazil) at 2000 rpm. The dentinal walls were prepared by a 3 mm long and 1.5 mm diameter ultrasonic tip. (CVD T0F-2; CVD-Vale, São José dos Campos, SP, Brazil), coupled on a device used to allow the parallelism between the tip and root canal walls.

The ultrasonic device (CVD, CVD-Vale) was set at power 5, under irrigation. The preparation time was approximately 17 s (12). After preparation, cavities were irrigated with 5 mL saline. The samples were randomly divided (n=10) according to the retrofilling material: MTA Angelus (MTAA), MTA Sealer (MTAS, experimental), Sealer 26 (S26) and zinc oxide and eugenol cement (ZOE). The materials were placed in yhe cavities with a condenser (S.S.White, Rio de Janeiro, RJ, Brazil) until the complete filling and the samples were maintained at 37 °C for 48 h. After this period, 200- and 600-grit sandpaper were used to expose the material/dentin interface. For mechanical tests, each resin/dentin/root-end filling material disc was placed in the mechanical test machine (EMIC DL 2000), with a 5 kN load cell.

Progressive compression test was performed with the force applied from cervical to apical at 1 mm/min speed, from the contact of the device tip to root-end filling material displacement, as used in similar studies (1). The cylindrical tip had 1.3 mm diameter and maintained contact with sealer (15).

The values were obtained in newton (N) and transformed into MPa. To express the bond strength in megapascal (MPa), the recorded value was divided by the adhesion surface area of root canal filling, calculated by a specific formula. The area (mm^2) under load was calculated by the cylinder lateral surface area formula: bonding area=2πrh, where "r" is the radius of the preparation circumference, and "h" is the thickness of the root slice (2.0 mm). The push-out strength value in megapascal (MPa) was calculated by dividing the load (N) by bonding area (mm^2). Data were subjected to ANOVA and Tukey's tests at 5% significance level.

After the push-out bond strength test, each specimen was analyzed using a stereomicroscope at 20× magnification (Olympus SZ61, Olympus Optical Co., Tokyo, Japan). The failure modes were classified according to the following criteria: adhesive failure between sealer and dentin; cohesive failure within sealer and mixed failure when both failures were observed.

Results

The means (in MPa) and standard deviations obtained

Table 1. Composition and manufacturers of the used retrofilling materials

Material	Composition	Manufacturer
MTA Angelus	SiO_2,K_2O, Al_2O_3, NaO, Fe_2O_3, SO_3, CaO, Bi_2O_3, MgO and insoluble residues (silicate crystals, calcium oxide, potassium sulfate and sodium sulfate)	Angelus Indústria de Prdodutos Odontológicos Ltda, Londrina, PR, Brazil
MTA Sealer	White Portland cement, resin, calcium chloride, zirconium oxide (radiopaque filler)	Araraquara Dental School, UNESP, Brazil
Sealer 26	Powder: bismuth trioxide, calcium hydroxide, hexamethylenetetramine, titanium dioxide. Liquid: bisphenol epoxy.	Dentsply Indústria e Comércio LTDA, Petrópolis, RJ, Brazil
Zinc oxide and eugenol	Powder: zinc oxide, hydrogenated resin, bismuth subcarbonate, barium sulfate and sodium borate	S.S. White, Rio de Janeiro, RJ, Brazil

Table 2. Push-out bond strength means (MPa) and standard deviations

Group	Push-out bond strength
MTA Angelus	19.18^a (4.70)
MTA Sealer	19.13^a (2.65)
Sealer 26	15.91^a (3.82)
Zinc oxide and eugenol	9.50^b (3.73)

Means followed by different letters differ significantly.

Table 3. Percentage (%) of failure modes observed in each group after debonding

Failure mode	MTA Angelus	MTA Sealer	Sealer 26	Zinc oxide and eugenol
Adhesive	70	80	-	70
Cohesive	-	-	30	-
Mixed	30	20	70	30

are presented in Table 2. The bond strength to dentin walls was lower for ZOE ($p<0.05$) compared with the other materials. The most common failure mode was adhesive. For ZOE cement mixed failures were predominant (Table 3).

Discussion

Root-end preparation using ultrasonic tips contributed to increase endodontic surgery success rate (16). Root-end ultrasonic preparation provides better retention of retrofilling material and lower exposure of dentinal tubules (12,15). In the present study, root-end preparation was performed using a diamond ultrasonic tip (CVD T0F-2; CVD-Vale, São José dos Campos, SP, Brazil), which demonstrated cutting effectiveness and regular root-end preparation (12). Thus, to evaluate the root-end filling materials' bond strength, the dentinal walls were prepared by ultrasonic tips, which provide proper retrograde preparation (13).

The push-out test is widely used to evaluate the bond strength between the dentin and root-end filling materials (15). In the present study a device with a 1.3-mm tip diameter was used for preparation. The ultrasonic tip was coupled to an apparatus that allows parallelism between the tip and root canal walls.

Use of EDTA has not been recommended after retrograde cavity preparation in order to prevent exposure of a greater amount of open dentinal tubules in the apical surface after apicoectomy. Also, according to Celik et al. (17), irrigation regimes using EDTA have no effect on the push-out bond strength of the calcium silicate cements (17). The obtained results showed that MTA Angelus (19.18 MPa) and MTA Sealer (19.13 MPa) exhibited the highest bond strength values. Sealer 26 showed similar results to MTA-based materials. This material was used in a thicker consistency favoring its clinical application as root-end filling material (18,19). Amoroso-Silva et al. (18) also observed proper bond strength for Sealer 26 (24.80 MPa).

According to Hong et al. (20), the push-out strength was improved when MTA with calcium chloride was used. The results of the present study are consistent with those of Amoroso-Silva et al. (18), who found 25.35 MPa for MTA Angelus after push-out test using apical sections of roots from human teeth.

MTA Sealer composition is based on epoxy resin, which favors the sealing (21). MTA Sealer, besides containing resin and calcium chloride, contains white Portland cement, which may increase the adhesion due to its expansion (5). Furthermore, after longer periods, formation of either hydroxyapatite layer or carbonated apatite on its surface in contact with fluids containing phosphate could favor the chemical bonding between MTA and the dentinal walls (22). The results of the present study confirmed higher resin cements' bond strength compared with ZOE-based cements (23). Materials based on epoxy resin may present volumetric expansion contributing to bond strength of these materials (23). ZOE cement has lower bond strength values (9.50 MPa). The obtained results may be explained by the presence of zinc ion from zinc oxide, which may affect the mineral content of dentin (24).

Adhesive failures were predominant, agreeing with Shokouhinejad et al. (2). Those authors also observed a larger number of adhesive failures in MTA and associated this failure mode to the material displacement caused by its high compressive strength (2). However, ZOE cement showed predominance of mixed failures. This failure mode could be related to the low compressive strength of ZOE-based cements, compared to MTA-based cements and calcium silicate cements (25).

Based on the methodology and obtained results, it may be concluded that MTA Sealer (experimental), MTA Angelus and Sealer 26 after ultrasonic root-end preparation presented higher bond strength to dentinal walls than ZOE.

Resumo

O objetivo deste estudo foi avaliar a resistência de união de materiais retrobturadores. Quarenta fatias de 2 mm de espessura foram obtidas a partir de dentes unirradiculares humanos. Após o preparo do canal radicular usando uma broca cilíndrica de 1,5 mm de diâmetro, as paredes de dentina foram preparadas usando uma ponta de ultra-som diamantada (CVD T0F-2). As amostras foram divididas de acordo com os materiais (n=10): MTA Angelus (MTAA), MTA Sealer (MTAS, experimental), Sealer 26 (S26) e óxido de zinco e eugenol (ZOE). O teste de push-out foi realizado utilizando uma máquina de ensaios mecânicos (EMIC DL 2000) com velocidade de 1 mm/min. O tipo de falha foi avaliado em estereomicroscópio. Os resultados foram submetidos a ANOVA e teste de Tukey, com níveis de variância de 5%. MTAA (19,18 MPa), MTAS (19,13 MPa) e S26 (15,91 MPa) apresentaram os maiores valores de resistência de união ($p<0,05$). ZOE (9,50 MPa) apresentou os menores valores de resistência de união ($p<0,05$). A falha adesiva foi prevalente em todos os grupos, com exceção do ZOE, que apresentou falhas mistas. Concluiu-se que os materiais retrobturadores MTA Angelus, MTA Sealer e Sealer 26 apresentam maior resistência de união às paredes dentinárias que o óxido de zinco e eugenol após o preparo retrógrado.

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