Push-out bond strength of Calcium hydroxide and Mineral Trioxide Aggregate based sealers to root canal dentin

Resistência adesiva de cimentos endodônticos a base de Hidróxido de Cálcio e Agregado Trióxido Mineral na dentina radicular

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Abstract

Purpose: The adhesiveness of six root canal sealers: Acroseal, Endo CPM, Epiphany, White MTA, Sealapex and Sealer 26 to dentin, was evaluated in a push-out test design.

Methods: Twenty eight roots of freshly extracted teeth were gauged with a size 5 Largo drill. With a cutting machine slices of 2 mm were prepared, rinsed with 5.25% NaOCl and a final rinse with 17% EDTA, dried and filled with one of the sealers. After setting their bond strength was measured in a mechanical testing machine. The data were statistically analyzed by using a One Way ANOVA and post hoc Tukey test.

Results: The mean and standard deviation from values of bond strength was: Sealapex 2.2±0.4; Endo CPM 3.8±1.3; White MTA 6.0±1.4; Epiphany 10.9±2.6; Sealer 26 12.3±2.3; and Acroseal 12.2±1.4. Acroseal, Sealer 26 and Epiphany presented a significantly ($P<0.01$) greater bond strength compared with the other sealers. Also White MTA showed higher adhesiveness compared with Endo CPM and Sealapex ($P<0.01$).

Conclusion: The bond strength between endodontic sealers and root dentin was maximal when Acroseal, Sealer 26 and Epiphany were used; Sealapex e Endo CPM, in turn, presented the lowest bond strength mean values.

Key words: Endodontics; dental bonding; root canal filling materials

Resumo

Objetivo: Avaliar a adesividade de seis cimentos endodônticos à dentina radicular (Acroseal, Endo CPM, Epiphany, MTA branco, Sealapex e Sealer 26).

Metodologia: Vinte e oito dentes morradiculares foram preparados com uma broca largo #5. Fatias com 2 mm de espessura foram preparadas, irrigadas com NaOCl 5.25% e com EDTA 17% EDTA, secadas e obturadas com um dos cimentos testados. Após tomarem presa, a resistência adesiva dos cimentos à dentina radicular foi mensurada em uma máquina de ensaios universal. Os dados foram analisados estatisticamente usando ANOVA 1-fator e teste de Tukey.

Resultados: O Acroseal (12.2±1.4), Sealer 26 (12.3±2.3) e Epiphany (10.9±2.6) apresentaram maior resistência adesiva que os demais cimentos ($P<0,01$). Ainda, o MTA branco (6.0±1.4) apresentou maior adesividade quando comparado com o EndoCPM (3.8±1.3) e o Sealapex (2.2±0.4) ($P<0,01$).

Conclusão: A composição dos cimentos endodônticos influencia a adesão destes à dentina radicular. A resistência adesiva alcançada pelos cimentos Acroseal, Sealer 26 e Epiphany à dentina radicular foi superior àquela obtida pelo MTA branco, Endo COM e Sealapex.

Palavras-chave: Endodontia; adesão dentária; materiais restauradores do canal radicular

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Introduction

The objectives of the root canal filling are to prevent leakage from both the oral cavity and the periradicular tissues into the root canal system and to seal inside the root canal any irritant that could not be removed during the cleaning and shaping procedures (1). One of the main purposes of root canal treatment is the three-dimensional filling of the cleaned and shaped root canal (2).

The standard method for obturation of the root canal system uses a core material in combination with a root canal sealer. Gutta-percha cones and endodontic sealer are the most widespread materials used for this purpose. Since gutta-percha cones do not bond to dentin, the use of an endodontic sealer is necessary in order to provide an adequate and long-lasting seal of the root canal filling (3). For this reason, the attention of researchers is currently focused not only on the complete filling of the root canal space but also on the adhesion of this dense mass to the tooth structure (4,5). Adhesion of a root canal sealer is defined as its capacity to attach to the dentinal walls of the root canal and provide bonding between it and gutta-percha points; additionally, the sealer must also have cohesive strength to hold the obturation together (6). This adhesive property of the sealer reduces the empty space that allows the percolation of fluids into the interface, and later in the manipulation of this canal, it may help to maintain the root canal filling in place, especially if an intracanal retainer is needed (7).

Nowadays, researchers are looking for materials that present both biological and physical qualities. In this scenario, endodontic sealers composed of biocompatible materials have increased in endodontics. Calcium hydroxide ($Ca[OH]_2$) is the most widely used and investigated intracanal medicament because of its antibacterial and biological properties (8). For these reasons, endodontic sealers have been developed with $Ca(OH)_2$ as a component (e.g., Sealer 26, Dentsply Maillefer, Ballaigues, Switzerland; Sealapex, SybronEndo Corporation, Orange, CA, USA; and Acroseal, Septodont, Sant-Maur-des Fossés, Cedex, France).

Mineral trioxide aggregate (MTA) is another material widely used in endodontics, mainly for sealing of perforations, pulp capping, pulpotomy, and root-end filling. Additionally, MTA has been suggested for root canal filling. MTA-based sealer Endo CPM (EGEO S.R.L., Buenos Aires, BA, Argentina) was developed for root canal obturation. The composition of Endo CPM is similar to MTA plus barium sulfate and calcium chloride (5,9).

Alternatively, in 2002 the Resilon/Epiphany system (Pentron Clinical Technologies, Wallingford, CT, USA) was developed as an innovative resin-based cement. This system combines the use of adhesive and cement in a single application. In addition, an important point to consider is that the self-adhesive resin cement can bond to dentin without any kind of conditioning or pretreatment (10). Epiphany contains UDMA, PEGDMA, EBPADMA, and BISGMA resins, methacrylate, and $Ca(OH)_2$ in its formulation (4).

Thus, the purpose of this in vitro study was to compare the bond strength of $Ca(OH)_2$- and MTA-based sealers to root dentin using a push-out test design. Based on previous reports, the tested hypothesis was that $Ca(OH)_2$-based sealers would present higher bond strengths than MTA-based sealers.

Materials and Methods

The study was approved by the Ethical Committee of the Araraquara School of Dentistry. A total of 28 single-rooted freshly extracted human teeth were used in this study. All teeth were disinfected by complete immersion in 5% sodium hypochlorite for 2 h and were then stored in distilled water and frozen. Before root canal preparation, the crown was removed at the cementoenamel junction by using a high-speed carbide bur under water-cooling.

The working lengths were established by inserting a #15 K-file (Dentsply Maillefer, Tulsa, OK, USA) to the root canal end and subtracting 1 mm. The size of the root canal was measured by using Gates Glidden drills #1, #2, and #3, and Largo drills #1, #3, and #5 (ISO 150) to ensure that all canals were included in the preparation. During the instrumentation, the canals were irrigated with 1 mL of 5.25% sodium hypochlorite after each drill change. A final rinse with 3 mL of 17% ethylene diamine tetra-acetic acid (EDTA) for 5 min followed by irrigation with 3 mL of saline was performed to remove the smear layer.

Each root was then included in a polyester resin and sliced perpendicular to the long axis using a low speed saw (Isomet 1000 Precision Saw, Buehler, Lake Bluff, IL, USA) under water cooling. This created 4 to 5 slices that were 2-mm thick for each root sample. The thickness of each root slice was measured by means of a digital caliper. Every slice was marked with a number from the coronal end to the apical end. The number 1 was the coronal slice and number 5 the apical. This mark was made on the upper face of the slice. After a new irrigation with 3 mL of 17% EDTA, the slices were randomly allocated in flasks numbered from 1 to 6. These flasks were randomly assigned to each tested sealer (Sealapex, Endo CPM, White MTA, Epiphany, Sealer 26, and Acroseal). Manufacturers and the composition of cements are shown in Table 1.

Each group received 20 slices. All sealers were mixed according to the manufacturer's instructions and then inserted into the root canals. In the Epiphany group, the sealer was light-cured for 40 s from the coronal aspect. After the filling was completed, all samples were stored in a moist environment at 37°C for 48 h to allow the material to set. The shear bond strengths of the 6 groups were tested with a push-out technique by using a mechanical testing machine (MTT 808; Instron Corporation, Norwood, MA, USA). This process was accomplished by using a specially developed 1.3-mm diameter cylindrical stainless steel plunger and applying a constant compressive load at a speed of 1 mm/min until bond failure occurred. The plunger was positioned such that it only contacted the sealer and avoided contact

Table 1. Tested materials, composition and manufacturers.

Material	Composition	Manufacturers
Acroseal	Base Paste: 2 g glycyrrhetic acid, 25 g methenamine, radiopaque excipient q.s.p. 100 g Catalyst Paste: 28 g calcium hydroxide, 17 g bisphenol A diglycidyl ether, radiopaque excipient q.s.p. 100 g.	Specialités-Septodont, Saint Maur-des-Fossés, Cedex, França
Endo CPM	MTA: silicon dioxide, calcium carbonate, bismuth trioxide, barium sulfate, propyleneglycol alginate, sodium citrate, calcium chloride, active ingredients	EGEO S.R.L. Bajo Licencia MTM Argentina S.A., Buenos Aires, Argentina
Epipanhy	UDMA, PEGDMA, EBPADMA, BISGMA and methacrylate resins; barium borosilicate glasses treated with silane; barium sulfate; silica; calcium hydroxide; bismuth oxychloride with amines; peroxides; photopolymerization initiator; stabilizers and pigments	Pentron Clinical Technologies, LLC., Wallingford, CT, USA
White MTA	Silicon dioxide, potassium oxide, alumina, sodium oxide, iron oxide, sulfur trioxide, calcium oxide, bismuth oxide, magnesium oxide and insoluble residues (crystalline silica, calcium oxide and potassium sulphate)	Angelus, Soluções em Odontologia, Londrina, PR, Brazil
Sealapex	20% calcium oxide, 2.5% zinc oxide, 29% bismuth trioxide, 3% silicon particles, 20% titanium dioxide, 1% zinc stearate, 3% tricalcium phosphate, isobutyl salicylate + methyl salicylate + 39% pigment	SybronEndo – Sybron Dental Specialties, Glendona, CA, USA
Sealer 26	Powder: Bismuth trioxide, calcium hydroxide, hexamethylenetetramine and titanium dioxide Paste: Bisphenol epoxy resin	Denstply Indústria e Comércio Ltda., Petrópolis, RJ, Brazil

with the root canal walls. Also, the disk samples were positioned to allow the plunger to move in a single apical-to-coronal direction, which resulted in the displacement of the root filling toward the coronal part of the disk. The bond strength was determined by using a real-time computer software program, which plotted a load/time curve during compression testing. Bond failure load was noted when a sharp decline was observed on the graph and/or complete dislodgement of the root filling material. The bond strength was expressed in MPa by dividing the load in Newtons (N) by the area of the bonded interface (mm^2). Statistical analysis was performed using a one-way analysis of variance and the post hoc Tukey test, with significance set at $P<0.05$.

Table 2. Push-out bond strength (MPa) and standard deviations (SD) of the tested sealers.

Group	N	Mean* (MPa)	SD	Min.	Max.
Sealapex	20	2.2^{a}	0.4	1.60	2.97
Endo CPM	20	3.8^{a}	1.3	1.99	6.68
White MTA	20	6.0^{b}	1.4	4.11	8.63
Epiphany	20	10.9^{c}	2.6	6.26	14.62
Sealer 26	20	12.3^{c}	2.3	9.81	16.41
Acroseal	20	12.2^{c}	1.4	10.63	14.91

* The same lowercase letters were not significantly different ($P>0.05$).

Results

Table 2 presents the results obtained after the push-out bond strength test for the 6 studied sealers. Statistical analysis revealed significant differences ($P<0.01$) between sealers, which allowed them to be ranked in decreasing adhesiveness values. Acroseal, Sealer 26, and Epiphany presented the highest bond strengths with no statistically significant difference among them ($P>0.05$). Endo CPM Sealer and Sealapex showed the worst results ($P<0.01$), without any significant differences between them ($P>0.05$). Finally, White MTA presented intermediate results for bond strength.

Discussion

Efforts have been made to develop new endodontic sealers with great physical and biological properties. However, heretofore there is no endodontic sealer capable of combining both characteristics. Regarding the physical properties, the sealing ability of endodontic sealers is extremely important. The main goal is to create a homogenous and gap-free interface between the filling material and root dentin to prevent microleakage (11). For this reason, the aim of this study was to compare the bond strength of $Ca(OH)_2$- and MTA-based sealers to root dentin.

Several static (push-out, pull-out, microtensile) tests have been used to evaluate bond strength. However, the push-out test presents advantages such as a fewer number of premature failures, lower standard deviation, and that it is easier to perform (12). The utilization of root discs has some advantages when compared with other dentinal surfaces: the sealer is placed in contact with the dentinal surface inside the root canal instead of a flat surface from the crown region, which presents a different pattern of tubules (13). When the sample is filled with sealer, it adapts to the shape of the canal and penetrates into the dentinal tubules, establishing a mechanical retention. On the other hand, it is evident that in vitro models cannot faithfully reproduce the clinical conditions because the scenario is well controlled; the root dentin is not uniform and may vary from one tooth to another (14). Moreover, in vitro models have usually tested single-rooted teeth, which are less complex and present few anatomical variations when compared with multi-rooted teeth (11). For these reasons, single-rooted teeth were used for push-out testing. The literature presents a large variation in the thickness of the dentin slices tested (thickness ranging from 0.6 mm to 7.0 mm) (14,15). In the present study, 2-mm-thick specimens were created because they generate a lower percentage of cohesive failures and their dimensions are easier to measure (smaller radius, bigger radius, and height) in comparison with 1-mm-thick specimens (16).

The tested hypothesis was partially confirmed since the results showed higher bond strength values in the $Ca(OH)_2$-based sealer groups (Acroseal, 12.2 MPa; and Sealer 26, 12.3 MPa) compared to the MTA-based sealers (Endo CPM, 3.8 MPa; and White MTA, 6.0 MPa). However, Sealapex, a $Ca(OH)_2$-based sealer, presented the lowest bond strength values. In general, the bond strength of root canal sealers reported in the literature is lower than the results of this study. There are some possible explanations for these differences in the results (4,13,17).

Although testing designs that use natural canal spaces have a pragmatic appeal for clinicians, they have severe limitations from a materials science perspective (18). The main downside is the thermoplastic nature of core materials used for root canal filling (e.g., gutta-percha and Resilon cones). These cones have the tendency to deform during testing (19) and generate erroneous results. This could have been responsible, in part, for the report that sealers tested in thin films were considerably weaker than when they were tested in bulk by eliminating the compliant core material from the canal space (5). Previous studies used gutta-percha or Resilon cone as the core material and presented lower bond strength values when compared with the present results (4,17,20). In the present study, root canals were filled only with sealer as described previously (11,13). This is not the standard procedure for root canal filling, but the use of core materials negatively affects the push-out bond strengths of root canal filling materials (21). In addition, due to the absence of adhesiveness between Resilon or gutta-percha cones to root dentin, if these materials touched the dentin wall it would interfere with the results (22). Lastly, the shear bond strengths of sealers to gutta-percha and to Resilon may be variable (4). Some failures can occur between the main gutta-percha cone and the sealer, and other failures can occur on the sealer–dentin interface (17). These variations could also interfere in the results of the push-out test and were avoided in the present study.

Endo CPM Sealer is a MTA-based sealer that contains calcium chloride (23). The addition of this component improves the handling properties of MTA, increases the release of calcium ions (23), and acts as a setting accelerator (7). In the present study, White MTA presented higher bond strength than Endo CPM Sealer (6 MPa and 3.8 MPa, respectively). So it can be speculated that this component may affect the adhesion of MTA. Additionally, Endo CPM Sealer presents low flow properties, which might have an effect on the homogeneity of root canal fillings and consequently also on bond strength values (7).

Previous investigations have reported lower bond strength values for Epiphany than for non-bonded resin sealers (4,17,24). Even though the present study found higher bond strength values for Sealer 26 and Acroseal in comparison with Epiphany, the differences were not significant ($P>0.05$). One potential explanation for the superior adhesiveness to root dentin shown by epoxy resin-based sealers could be based on the creation of a covalent bond by an open epoxide ring to exposed amino groups in the collagen network (20). In addition, a higher amount of gaps between root dentin and Epiphany was reported (25). These gaps could act as a pathway for microorganism leakage and adversely affect the adhesion between sealer and root dentin. Acroseal and Sealer 26 present similar chemical compositions, which explain the similar bond strength values obtained in the present study.

Despite the differences in methodology, the results of the present study are in agreement with other studies showing that Epiphany is not superior to other resin-based sealers with regard to its adhesiveness to dentin (4,17,24). Further, we found that the resin-based sealers showed superior adhesiveness and Sealapex showed poor adhesive performance (9).

Conclusions

Within the limits of this study and the specific experimental conditions, it may be concluded that the composition of the endodontic sealers influences their bond strength to root dentin. The interfacial strength achieved using Acroseal, Sealer 26, and Epiphany to root dentin was superior to that achieved using White MTA, Endo CPM Sealer, and Sealapex.

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