
Adhesion of four endodontic sealers to root dentin conditioned with different chemical solutions

Adesão de quatro cimentos endodônticos em dentina condicionada com diferentes soluções químicas

Ceci Nunes Carvalho¹, Patrícia Guerreiro Monteiro¹, Eduardo Akisue¹, José Bauer², Antonio Carlos Bombana¹

¹School of Dentistry, University of São Paulo, São Paulo-SP; Brazil; ²School of Dentistry, Federal University of Maranhão, São Luis-MA, Brazil.

Abstract

Objective – To evaluate *in vitro* adhesion of endodontic sealers Rickert, AH Plus, Sealapex and Epiphany to dentin previously conditioned with one of two different chemical solutions. **Methods** – A total of 80 specimens were made up with bovine incisive teeth that were prepared by cutting predetermined sections and wearing them, producing standard experimental samples. Forty samples were treated with 2.5% sodium hypochlorite and divided into four groups with 10 specimens each, to be coated with the different sealers. Dentin of other forty samples was treated with 17% EDTA, and these samples were also divided into four groups. Prepared specimens were hydrated uniformly, dried, bonded and kept at 37°C and 100% relative humidity. After 72 hours, specimens were analyzed using an Instron universal testing machine in traction mode. Data were analyzed by Kruskal-Wallis test ($\alpha=0.05$). **Results** – EDTA treatment led to the greatest adhesiveness in all experimental groups. **Conclusion** – It was concluded that conditioning of the surface with chelating agents increase adhesiveness of the tested sealers and similar behavior was observed in the adhesion of epoxy-based and methacrylate-based sealers.

Descriptors: Dental cements; Resin cements; Edetic acid; Root canal filling materials

Resumo

Objetivo – Avaliar *in vitro* a adesão dos cimentos endodônticos Rickert, AH Plus, Sealapex e Epiphany à dentina previamente tratada por duas soluções irrigadoras. **Métodos** – Foram empregados 80 corpos de prova obtidos a partir de dentes incisivos bovinos que foram preparados por cortes e desgastes preestabelecidos, resultando em modelos experimentais padronizados. Quarenta dessas amostras foram tratadas com hipoclorito de sódio 2,5% e subdivididas em quatro grupos de 10 espécimes para cada cimento. As demais 40 amostras tiveram a dentina tratada com EDTA a 17% e foram divididos igualmente em 4 grupos. Os espécimes preparados foram uniformemente hidratados, secos, receberam os cimentos, e foram mantidos sob umidade relativa a 37°C. Após 72 horas os espécimes foram submetidos a testes de tração com auxílio da máquina de ensaio universal Instron, sendo os dados analisados pelo teste de Kruskal-Wallis ($\alpha=0.05$). **Resultados** – O tratamento com EDTA proporcionou maiores índices de adesão para todos os grupos experimentais ($p<0.05$). **Conclusão** – Pode-se concluir que o tratamento da superfície com agente quelante, aumenta a capacidade de adesão dos cimentos testados. Não houve diferença na adesão dos cimentos a base de resina epóxica e a base de metacrilatos.

Descritores: Cimentos dentários; Cimentos de resina; Ácido edético; Materiais restauradores do canal radicular

Introduction

Sealing of the root canal is an extremely important step for the outcomes of endodontic treatment. Because of that, new sealers have been developed in order to achieve maximum sealing of the root canal system.

Among the physical and chemical characteristics of importance in sealers used in restorative dentistry, adhesion has a strong relationship with marginal sealing capacity¹⁻².

Zinc oxide/eugenol-based sealers (Rickert and Sealapex) and epoxy resin-based sealers (AH Plus) have been largely used in dental practice and show adequate rates of success and sealing capacity¹⁻².

Different from the classic concept of using gutta-percha associated with a sealer, methacrylate-based sealers have recently been developed in order to seal the endodontic space with dual-cure composite resins. This new concept has been proven to be efficient in terms of marginal sealing³⁻⁷.

Increased adhesion depends on the area of contact⁸, which can be increased by using demineralizing agents⁹⁻¹⁰. Ethylenediaminetetraacetic acid (EDTA) solution in different concentrations is able to remove the smear layer and expose a large number of dentinal tubules¹¹⁻¹⁴.

Therefore, the objective of this study was to evaluate the adhesiveness of four endodontic sealers (Rickert, Epiphany, Sealapex and AH-Plus) to dentin treated with one of two conditioning regimens (2.5% sodium hypochlorite – and 17% EDTA) as determined by the traction strength necessary to break the sealer/dentin interface.

Methods

Eighty experimental samples were made from lower incisive bovine teeth. After teeth were cleaned, incisal surface was worn until all enamel was removed, leaving a flat surface of exposed dentin. Proximal, vestibular and lingual surfaces were worn with diamond disks and carbide cylindrical burs until a 7.5 x 4 mm surface (Figure 1A), measured with a digital caliper Mitutoyo 500-143B, was obtained.

Prepared teeth had their roots fixated in acrylic resin and were placed inside a PVC ring, keeping the crown exposed and the prepared surface parallel to the horizontal plan, as determined by a bubble level (Figure 1A).

All surfaces were flattened in 0.3 mm using a hard tissue microtome (Labcut 1010) in order to produce parallel surfaces and a final area of 6.9 x 3.4 mm.

Chart 1. Experimental groups

Experimental groups	
Conditioning with 2.5% sodium hypochlorite (F&A Lab. Ltda., São Paulo-SP, Brazil)	Conditioning with 17% EDTA (F&A Lab. Ltda., São Paulo-SP, Brazil)
Group RKSH (n=10) Rickert (F&A Lab. Ltda., Sao Paulo, SP)	Group RKED (n=10) Rickert
Group EPSH (n=10) Epiphany (SybronEndo Corp, Orange, USA)	Group EPED (n=10) Epiphany
Group SLSH (n=10) Sealapex (SybronEndo Corp, Orange, USA)	Group SLED (n=10) Sealapex
Group AHSH (n=10) AH Plus (Dentsply DeTray GmbH, Konstanz, Germany)	Group AHED (n=10) AH Plus

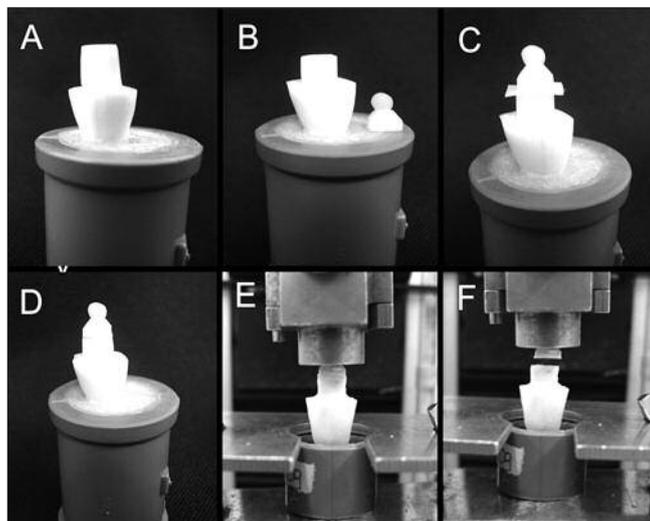


Figure 1. Sequence of the preparation of samples for traction assay

All surfaces were conditioned with 35% phosphoric acid and treated with an adhesive Adper Single Bond 2. A metal matrix was used to create the composite resin truncated-cone (Figure 1B) to be fixed in the traction handle of the universal testing machine.

Specimens were placed again in the hard tissue microtome and a cut was made 2mm below the resin truncated-cone to create incisal counter surfaces (Figure 1B).

To clean and remove the smear layer produced by the cuts, specimens and their respective incisal counter surfaces were ultrasonicated in a BioSonic UC50DB unit for 10 minutes. After that, they were hydrated in saline solution for 72 hours at 37°C. After hydration, specimens were randomly divided into 8 experimental groups according to the sealer and treatment used (Chart 1).

Treatment of dentinal surfaces with 2.5% NaOCl (groups 1, 2, 3 and 4) and 17% EDTA (groups 5, 6, 7 and 8) was carried out by dripping the conditioning solution on the dentin at a constant flow of 4 mL/min. After conditioning, the space between the incisal surface and counter surface was dried twice for 30 seconds (Figure 1C).

Sealers were prepared according to the manufacturers' instructions and placed in the space between the incisal surface and counter surface (Figure 1D). A weight of 19.6 N (2 Kgf) was applied to the samples in order to achieve uniform pressure in all experimental groups.

Experimental groups 1, 3, 4, 5, 7 e 8 were kept for 72 hours at 37°C and 100% relative humidity. Groups 2 and 6, treated with Epiphany sealer, were kept in the same conditions but under vacuum, once the presence of oxygen affects dual cure.

After final setting, traction assays were carried out in a universal testing machine Instron model 3342, at a speed of 0.5mm/min (Figures 1E and 1F). Values in Newtons (N) were recorded for statistical analysis.

Results

Table 1 showed means and standard deviations in Newtons (N) for each experimental group.

Original data showed non-normal distribution and statistical analysis was based on Kruskal-Wallis non-parametric test followed by Student-Newman-Keuls comparative test.

When sealers were compared, no statistically significant difference was found between the two conditioning treatments (Table 2), but there was a significant difference in favor of the demineralizing solution in all experimental groups ($p < 0.05$).

Table 1. Means and standard deviation of traction strength of the experimental groups (N)

Groups	Means and standard deviation (N)
RKSH – Rickert + NaOCl	4.69 ± 0.60
EPSH – Epiphany + NaOCl	9.32 ± 1.58
SLSH – Sealapex + NaOCl	1.43 ± 0.44
AHSH – AH Plus + NaOCl	62.73 ± 10.80
RKED – Rickert + EDTA	11.01 ± 2.10
EPED – Epiphany + EDTA	15.68 ± 2.54
SLED – Sealapex + EDTA	1.92 ± 0.57
AHED – AH Plus + EDTA	78.1 ± 3.37

Table 2. Statistical differences between experimental groups (p value)

Comparison	Conditioning with NaOCl	Conditioning with EDTA
Rickert vs. Epiphany	0.1814	0.2617
Rickert vs. Sealapex	0.1814	0.1490
Rickert vs. AHPlus	0.0075	0.0103
Epiphany vs. Sealapex	0.0075	0.0103
Epiphany vs. AHPlus	0.1814	0.1490
Sealapex vs. AHPlus	<0.0001	<0.0001

Discussion

Dentinal adhesion used in endodontics has been considerably improved in the last few years in relation to marginal sealing of the endodontic system¹⁵⁻¹⁷.

Recently, a new endodontic obturation system was created to replace gutta-percha and conventional sealers. RealSeal (SybronEndo) or Epiphany (Pentron Clinical Technologies) follow the same concept of adhesion of restorative dentistry based on an auto-conditioning primer and a methacrylate-based hydrophilic sealer asso-

ciated with a core made with thermoplastic synthetic polymer. The objective of this association of materials is perfect adhesion between core/sealer/dentin interfaces, producing an ideal monoblock and adequate marginal sealing³⁻⁷.

Increased adhesiveness of endodontic sealers depends on the contact surface area⁸, which can be increased by removing the smear layer with demineralizing solutions⁹⁻¹⁰. EDTA is commonly chosen due to its chelating capacity and is widely used as a conditioning solution in the preparation of the root canal^{11-14,18-19}. As previously observed by Pécora *et al.*¹⁹ (2001), Haragushiku²⁰ (2010) and Nunes *et al.*²¹ (2008), the use of EDTA improved adhesiveness in all experimental groups ($p < 0.05$).

When using methacrylate-based sealers, NaOCl could interfere in polymerization reactions caused by the release of free radicals, as it is the case in dual-cure cements, where residual oxygen negatively affects their polymerization²²⁻²³. This may be the explanation for the lower results ($9.32N \pm 1.58N$) obtained in the methacrylate sealer group compared with the group that was conditioned with the chelating solution ($15.68N \pm 2.54N$).

In order to prevent interference of oxygen in the polymerization of the dual-cure sealer (Epiphany) immediately after sealing, specimens were photopolymerized and stored under vacuum for 72 hours.

Results showed a wide variation in the adhesiveness of the different sealers. The lowest values were observed for zinc/eugenol-based sealers: Sealapex showed the lowest adhesiveness compared with resin-based sealers AH Plus ($p < 0.0001$) and RealSeal ($p = 0.0075$), and adhesiveness similar to that of Rickert ($p = 0.1814$).

In the same analysis, resin-based sealer AH Plus was statistically different from Rickert ($p = 0.0075$) and Sealapex ($p < 0.0001$), but did not from methacrylate-based sealer RealSeal ($p = 0.1814$). These findings were similar to those of Nunes *et al.*²¹ (2008), who did not observe differences ($p < 0.01$) between AH Plus ($0.78kN \pm 0.13$) and Epiphany ($0.61kN \pm 0.19$). However, these authors showed differences between surface treatments (distilled water: $0.58kN \pm 0.19$; 1% NaOCl: $0.71kN \pm 0.12$; 1% NaOCl+17% EDTA: $0.80kN \pm 0.17$; $p < 0.05$). This greater adhesiveness observed in both studies may be explained by the hydrophobic characteristics of this sealer, that prevent the negative influence of moisture on setting.

As for the experimental model, we chose to make specimens from bovine teeth due to the fact that they are more easily obtained, different from human teeth. Besides, bovine dentin is similar to human dentin in terms of permeability²⁴ and morphology of dentinal tubules²⁵.

Conclusion

Considering the importance of the subject and the large number of variables that may influence adhesion of new resin-based sealers, other studies should be done to determine the exact relationship of conditioning regimens in the final adhesiveness or marginal sealing.

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Corresponding author:

Ceci Nunes Carvalho
Rua Catalunha, 330
São Paulo-SP, CEP 05329-030
Brazil

Email: cecicar@usp.br

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