

Evaluation of Flow, and Dimensional Alterations of Epoxy Resin-Based Root Canal Sealers

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ABSTRACT - The aim of this study was to evaluate the epoxy resin-based cements AH Plus (AH) and Sealer 26 (S) compared with Endofill (E) in terms of flow, and dimensional alteration properties, based on ADA Specification No 57. For flow test, a volume of 0.5 ml of cement was placed on glass plate (40 x 40 x 5 mm) using a graduated syringe. At 180 ($\pm$ 5) s after the start of mixing, a load of 100 N plus the top plate with a mass of 20 ($\pm$ 2) g were placed on top of the material. Ten min after the commencement of mixing, the load was removed and the average of the major and minor diameters of compressed disc of material was recorded (both diameters must agree within 1 mm). The mean of five such determinations was taken as the flow of the material. For dimensional alterations, 12-mm high cylindric specimens measuring 6-mm in diameter were prepared and left to stand for a period corresponding to three times the setting time. These specimens were immersed in 30 ml of deionized distilled water after measuring their length with a caliper. Thirty days later, the sample was removed from the container, dried and measured again for length to determine the percent of dimensional alteration. The results showed statistical difference ($p < 0.01$) between the sealers in terms of both properties: flow: AH (40.31 mm), S (52.70 mm), and E (37.41 mm); dimensional alteration: AH (+0.23), S (+3.07 mm), and E (+0.09 mm). We concluded that flow and dimensional alteration of studied cements conformed to ADA standards.

KEYWORDS - Flow, dimensional alterations, endodontic sealers, physicochemical properties.

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Introduction

Successful root canal treatment requires a hermetic seal of the root canal system. It is known that the majority of endodontic failures have been caused by the incomplete sealing of the root canal (1), confirming the necessity of using materials with satisfactory properties. The properties of the root canal sealers can be classified as physicochemical, antimicrobial and biological. In 1984, a series of regulations and tests were made effective, created one year earlier by the American Dental Association (2) for the standardization of research of these properties.

Numerous root canal sealers are available, based on various formulas and containing a variety of different components, such as epoxy resin-based sealers, materials containing calcium hydroxide, and zinc oxide-eugenol-

based cements. The epoxy resin-based sealers, such as Sealer 26 is an AH26 derivative with calcium hydroxide added (3), and are based on bisphenol-A-diglycidylether. The powder contains hexamethylene tetramine, which is responsible for the setting reaction of that material with bisphenol-A-diglycidylether. Some studies related to physicochemical properties of Sealer 26 have been reported in the literature. Carvalho-Junior *et al.* (4) found that Sealer 26 promotes low values of solubility when compared with zinc oxide-eugenol-based cement.

AH Plus is a two component paste root canal sealer also based on epoxy resin introduced to substitute AH26. According to the manufacturer descriptions, AH Plus has the advantageous properties of AH26, but preserves the chemistry of the epoxy amines better so that the material no longer releases formaldehyde, that interferes

negatively in the biocompatibility of AH26 (5). Pécora *et al.* (6) investigated the adhesiveness of AH Plus comparing with zinc oxide-eugenol-based cement, and other epoxy resin-based sealers, and they verified the superiority of that material.

The zinc oxide-eugenol-based cement was introduced in endodontics in 1936 by Professor L.I. Grossman (7). In 1974, he concluded his researches and this endodontic filling cement has been used for years with satisfactory physicochemical (8) and antimicrobial (9) properties.

In this study, we have compared the epoxy resin-based cements AH Plus and Sealer 26 compared with zinc oxide-eugenol-based cement Endofill in terms of flow, and dimensional alteration properties, based on ADA Specification No 57.

Material and Methods

The materials studied were AH-Plus (Dentsply DeTrey, Konstanz, Germany), Endofill (Dentsply-Herpo, Petrópolis, RJ, Brazil), and Sealer 26 (Dentsply-Brasil, Petrópolis, RJ, Brazil). The flow, and dimensional alterations of the cements were determined according to ADA Specification 57 (American National Standards Institute, 1984) recommendations.

The powder/liquid ratio and the spatulation time were determined by the method of Sousa-Neto *et al.* (10) (Table 1).

Flow

For flow test, a volume of 0.5 ml of cement was placed on glass plate (40 x 40 x 5 mm) using a graduated syringe. At 180 ($\pm$ 5) s after the start of mixing, a load of 100 N plus the top plate with a mass of 20 ($\pm$ 2) g were placed on top of the material. Ten min after the commencement of mixing, the load was removed and the average of the major and minor diameters of compressed disc of material was recorded.

Two conditions were necessary to validate the tests: the difference between the largest and the smallest diameters could not exceed 1.0 mm and the disc should be uniformly circular. If these conditions were not found, the test was repeated following the same experimental protocol. Five repetitions were carried out for each cement and the arithmetic mean was obtained, representing the flow of the tested material.

Dimensional Alterations

Three Teflon molds were prepared for the manufacture of 12-mm high cylindric samples measuring 6 mm in diameter. The mold was placed on a 1-mm thick, 25-mm wide, and 75-mm long glass plate wrapped with a fine cellophane sheet. The mold was then filled until a slight excess of material was observed at its upper end. A microscope slide also wrapped in cellophane was then pressed onto the upper surface of the mold. The assembled microscope slide, cellophane-wrapped slide, and the mold containing the material in the middle were then kept firmly joined with the aid of a C-shaped clamp. Five minutes after the mixture was first prepared, the assembly was transferred to a chamber with 95% relative humidity and a temperature of 37°C. The assembly was left to stand for a period corresponding to three times the setting time and removed from the chamber.

The next step consisted of filling the ends of the mold containing the sample with distilled and deionized water to obtain a regular surface. The sample was then removed from the mold, measured for length with a caliper, and stored in a 50-ml vessel containing 30 ml of deionized distilled water at 37°C for 30 days. The sample was then removed from the container, blotted dry on absorbent paper, and measured again for length.

Percent dimensional alterations were calculated using the following formula:

Table 1. Powder/fluid ratios and spatulation times of the cements studied.

Sealer	Grams powder/0.20 ml liquid (range)	Mean (g)	Spatulation time (range, s)	Mean (s)
AH Plus	ND	ND	ND	ND
Endofill	0.99-1.06	1.03	111-123	117
Sealer 26	0.36-0.42	0.39	80-89	84

ND = not done.

$[(L_{30 \text{ days}} - L)/L] \times 100$, where $L_{30 \text{ days}}$ is the length of the sample after 30 days of storage under the experimental conditions and L is the initial length of the sample. The arithmetic mean of five replicates was recorded as the dimensional alteration of the cement tested.

Statistical Analysis

The Tukey test was used for statistical analysis ($p < 0.01$).

Results

Flow

Table 2 shows the mean values of the flow test for the 3 sealers. Statistical analysis showed significant differences ($p < 0.01$) between the tested sealers, except for AH Plus and Endofill, that were statistically similar. The sealers can be ranked in decreasing values of flow: Sealer 26, AH Plus, and Endofill.

Dimensional Alterations

The dimensional alterations (Table 3) showed significant differences ($p < 0.01$) between the tested sealers, with higher dimensional alteration values for Sealer 26. The zinc oxide-eugenol-based cement Endofill revealed the lowest dimensional alteration values, and AH Plus cement was in an intermediate position.

Discussion

The flow of a root canal sealer is an important factor for clinical performance of the material, because it reflects its capacity to

penetrate into small irregularities of dentin and also into lateral canals (8). For testing the flow, ADA specification number 57 recommends the formation of a disc of at least 25 mm in diameter by the sealer. As can be seen in Table 2, all sealers met this condition. AH Plus cement presented flow average value of 40.31 mm. Endofill cement presented an average value of 37.41 mm. Sealer 26 cement showed the highest flow value (52.70 mm).

Endofill, a zinc oxide-eugenol-based sealer, has shown good impermeability, volume stability, adherence and dissolution (11,12). However, in this study, Endofill was the cement that presented the smallest flow values, among the studied cements. It can be explained by eugenol viscosity. Sousa-Neto (13) reported that liquid viscosity influences the flow of a sealer, and the most viscous liquid presents larger adhesion and cohesion forces among the molecules and, therefore, it promotes smaller flow of the material.

According to the manufacturers (Dentsply), AH Plus and Sealer 26 are epoxy resin-based sealers however they present different formulations. The difference between them lays in the fact that AH Plus is produced by De Trey, a Dentsply subsidiary in Germany, and Sealer 26 is produced by Dentsply-Brasil in Brazil. Sealer 26 has a formula similar to AH Silver Free (Dentsply), except that Sealer 26 has calcium hydroxide in its formulation while AH Silver Free does not. This addition of calcium hydroxide to the formula implies a reduction of the bismuth trioxide percentage (6). Sealer 26 and AH-Plus cements consist of a powder and liquid component and a paste/

Table 2. Flow (mm) of the cements studied.

Sealer	Flow (range)	Mean (%)
AH Plus	37.20-38.35	37.41
Endofill	39.01-41.62	40.31
Sealer 26	52.28 to 53.58	52.70

Table 3. Percent dimensional alteration of the cements studied.

Sealer	Dimensional alteration (range)	Mean (%)
AH Plus	+0.18 to +0.37	+0.23
Endofill	+0.09 to +0.17	+0.09
Sealer 26	+2.00 to +4.31	+3.07

+ = expansion, - = contraction.

paste component, respectively. Sealer 26 uses calcium hydroxide in the attempt of joining the characteristics of that agent to the powder of the cement and a bisphenol-A-diglycidylether epoxy resin as the liquid or gel. The powder and liquid systems of Sealer 26 allows the clinician to choose the viscosity of the material.

AH-Plus is sold as a formaldehyde-free cement on which the manufacturer has set the viscosity of the material. The paste A of AH-Plus endodontic filling cement contains an epoxy resin and iron oxide, and paste B contains amines and silicone oil. Both paste A and paste B have calcium tungstate, zirconium oxide, and aerosil. AH-Plus was developed by the manufacturer to display better technical, clinical, and cytotoxic characteristics than the original AH 26 epoxy material (14).

AH26 and AH Plus are basically the same material. The difference between them lies in the presence of silicone and aerosil in the formula and elimination of formaldehyde release from the latter material (15).

In spite of the AH Plus cement to be a two paste system cement, with low viscosity, the rheological characteristic, the size, and the form of the load particles of this cement might have influenced in the smallest performance when compared to the Sealer 26 cement for the flow test. Another aspect that can be speculated on the high value of flow of Sealer 26 is the amount, in volume, of bisphenol-A-diglycidylether free, that did not participate in the setting reaction with hexamethylenetetramine that is contained in the powder.

The dimensional alteration test recommended by ADA Specification 57 evaluates dimensional alterations of filling materials after experimentation. ADA Specification 57 recommends that no cement should present contraction higher than 1%. Therefore, all cements studied presented little expansion, are according to the followed specification.

Endofill presented expansion, probably, because of the presence of gum rosin in their formulas, which according to Sousa-Neto *et al.* (10), is responsible for cement expansion.

AH Plus and Sealer 26 presented expansion although the epoxy resin promotes polymerization contraction. AH Plus cement

presented little expansion (0.23%), when compared to Sealer 26 (3.26%), which may be related to ADA methodology that recommends that a sample be immersed in water after a period of three times the setting time of the material, in other words, after polymerization reaction. According to Phillips (1991), this dimensional alteration could be explained by water sorption suffered by these types of resins after their polymerization.

The contraction of root canal sealers must be as small as possible so that it can promote a hermetic sealing, thus favoring clinical success, because microleakage may occur from the cervical to apical third or inversely.

We conclude that all cements studied are according to ADA Specification 57 for the dimensional alteration property. In terms of flow, all cements also were according to ADA recommendations, with Endofill presenting the lowest flow values.

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Resumo

ARRUDA, Marcos Pôrto de, CARVALHO-JUNIOR, Jacy Ribeiro de, GENTIL, Marcelo, SOUSA-NETO, Manoel D., SOUZA-FILHO, Francisco José de, e CORRER-SOBRINHO, Lourenço. **Avaliação do Escoamento e Estabilidade Dimensional de Cimentos à Base de Resina Epóxica.** Oral Sci., mai./ago. 2005, vol.1, no.1, p.9-14.

O objetivo deste trabalho foi avaliar o comportamento dos cimentos à base de resina epóxi AH Plus (AH) e Sealer 26 (S) comparado com o cimento Endofill (E), em relação às propriedades de escoamento e estabilidade dimensional, com base na Especificação nº 57 da ADA. Para o escoamento, um volume de 0,5 ml de cimento foi colocado sobre uma placa de vidro (40 x 40 x 5 mm) usando uma seringa graduada. Após 180 ($\pm$ 5) s do início da mistura, uma carga de 100 N juntamente com outra placa de vidro com uma massa de 20 ($\pm$ 2) g foram colocadas sobre o material. Dez minutos após o início da mistura, a carga foi removida e a média do maior e menor diâmetro do disco de material comprimido

foi registrado (a diferença entre ambos diâmetros não podem ser superiores a 1 mm). A média das cinco determinações foi tida como o escoamento do material. Para estabilidade dimensional, confeccionaram-se corpos de prova cilíndricos dos cimentos, com dimensões de 12 mm de altura por 6 mm de diâmetro, passado 3 vezes o tempo de endurecimento do cimento. Após mensuração de seus comprimentos com um paquímetro digital, esses foram imersos em 30 ml de água destilada. Decorrido trinta dias, o corpo de prova foi removido, seco e medido novamente. Com os valores do comprimento inicial e final, determinou-se a variação percentual que ocorre nos corpos de prova.

Os resultados apresentaram diferença estatística ($p < 0.01$) entre os cimentos em relação a ambas propriedades: escoamento: AH (40,31 mm), S (52,70 mm) e E (37,41 mm); alteração dimensional: AH (+0,23), S (+3,07 mm) e E (+0,09 mm). We concluded that flow and dimensional alteration of studied cements conformed to ADA standards. Concluiu-se que, em relação ao escoamento e à estabilidade dimensional, todos os cimentos encontram-se dentro das normas da ADA.

PALAVRAS-CHAVE: Escoamento, estabilidade dimensional, cimentos obturadores de canais radiculares, propriedades físico-químicas.

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