Evaluation of the physicochemical properties of an epoxy-resin based root canal sealer with iodoform

Avaliação das propriedades físico químicas de cimento à base de resina epóxica acrescido de iodofórmio

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Abstract

Purpose: The objective of this study was to evaluate the physicochemical properties of the epoxy-resin based root canal cement Sealer 26®, enhanced with different amounts of iodoform.

Methods: The properties of setting time, radiopacity, flow, solubility and dimensional stability were measured according to ANSI/ADA Specification 57 for sealing materials. The samples were divided into five groups: (S) Sealer®; (SI5) Sealer® + 5% iodoform; (SI7) Sealer®+7% iodoform; (SI10) Sealer®+10% iodoform; (SI30) Sealer®+30% iodoform. Five samples were prepared from each group, for each test. Statistical analysis was performed using the one-way ANOVA test and post hoc Tukey-Kramer test at 5% significance level.

Results: The radiopacity and flow test results comply with ANSI/ADA norms. The values for setting time, solubility and dimensional stability did not meet the standards demanded by ADA Specification 57.

Conclusion: The addition of iodoform to the Sealer 26® endodontic cement did not alter the property for radiopacity. However, it decreases the solubility and increases the values for setting time, flow, and dimensional stability.

Key words: Root canal filling materials; iodoformium; physicochemical analysis

Resumo

Objetivo: O objetivo deste estudo foi avaliar as propriedades físico-químicas do cimento endodôntico à base de resina epóxica Sealer 26®, acrescido de diferentes quantidades de iodofórmio.

Metodologia: As propriedades de tempo de presa, radiopacidade, escoamento, solubilidade e estabilidade dimensional foram medidas de acordo com a Especificação 57 ANSI/ADA para cimentos. Os espécimes foram divididos em cinco grupos: (S) Sealer®; (SI5) Sealer® + 5% iodofórmio; (SI7) Sealer®+7% iodofórmio; (SI10) Sealer®+10% iodofórmio; (SI30) Sealer®+30% iodofórmio. Cinco espécimes foram preparados de cada grupo, para cada teste. A análise estatística foi realizada usando o teste ANOVA de fator único e o teste Tukey-Kramer ao nível de significância de 5%.

Resultados: Os resultados dos testes de radiopacidade e de escoamento foram compatíveis com as normas ANSI/ADA. Os valores de tempo de presa, solubilidade e estabilidade dimensional não atingiram os padrões requeridos pela Especificação 57 da ADA.

Conclusão: A adição de iodofórmio ao cimento endodôntico Sealer 26® não alterou a propriedade de radiopacidade. Porém, diminui a solubilidade e aumenta os valores de tempo de presa, escoamento e estabilidade dimensional.

Palavras-chave: Cimentos endodônticos; iodofórmio; análise físico-química

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Introduction

The complete and tridimensional hermetic sealing of root canal systems prevents infection and reinfection due to penetration by bacteria and their toxins, and consequent failure of the endodontic treatment (1). During the filling of root canal systems, the endodontic cement plays an important role in providing and maintaining this sealing (2). Root canal cements are classified in relation to their composition as: cements in a calcium hydroxide base; zinc oxide and eugenol; glass ionomer, silicon and resin (3).

In 1983, the *American Dental Association* (ADA), under the title of Specification 57, established norms and tests (flow, thickness of the film, setting time, radiopacity, solubility, dimensional stability) for the evaluation of the physicochemical properties of root canal cements, intended to promote uniformity of results, which led researchers to conduct these tests with higher standards and scientific rigor.

The first resin-based cement was recommended by Schröeder (4), who proposed a cement in a resin epoxy base of bisphenol A. Since then, research has contributed to improving the quality of that type of cement, which has resulted in several proposals, among them the Sealer 26® (Dentsply Ind. Com. Ltda., Petrópolis, Brazil).

The literature has shown that Sealer 26® has a low degree of solubility (5), antimicrobial activity (6), alkaline pH (7), appropriate adhesion (8), excellent dimensional stability (9) and flow within the standards of the American Dental Association (ADA) (10,11). However, it has moderate radiopacity when compared to other cements (12).

Aiming at maximizing the physicochemical properties of a endodontic cement, some authors have suggested adding some materials such as calcium hydroxide (13) and iodoform (14). The mixture of calcium hydroxide and iodoform, in paste form, has been used successfully to control infections in necrotic root canals and as a support in restoring periapical health, including root perforations (15,16). In addition to these characteristics, the clinical availability of iodoform to the dentist and its high molecular weight makes it an appropriate material for use as a radiopaque agent (17).

Hence, the objective of this study was to evaluate the Sealer 26® cement, enhanced with different amounts of iodoform, in regard to the physicochemical properties of setting time, radiopacity, flow, solubility, and dimensional stability based on Specification 57 of the ADA (11).

Materials and Methods

The Sealer 26® epoxy-resin based root canal cement and iodoform (K-Dent – Quimidrol, Joinville, Brazil) were used to conduct this study.

Tests for setting time, radiopacity, flow, solubility and dimensional stability of the samples were performed according to ANSI/ADA Specification 57 (2000) (11) for root canal sealer materials.

Five groups (n=5) were formed by the experimental procedures, in which Sealer 26 was used as recommended by manufacture (Group S) or with addition of 5% (Group SI5), 7% (Group SI7), 10% (Group SI10) or 30% (Group SI30) of iodoform.

For the physicochemical tests, the arithmetic mean of five repetitions of each group was calculated and considered to be the test result.

Determination of the powder/liquid proportions

A scale was used to weigh 0.88 g of Sealer 26® cement powder and 0.12 g of the resin, which compose the system. Then, the powder was added to the resin in small increments until the cement was of ideal consistency: that is, until it formed a 2.54 cm string to connect the spatula to the glass plate, or until the mass of cement remained on the spatula for 10 to 15 seconds before sliding off. After being mixed, the powder remaining on the glass plate was weighed and subtracted from the initial weight, thus obtaining the amount of powder effectively used in the mixture.

Setting Time

Stainless steel cylindrical molds, with internal diameter of 10 mm and uniform thickness of 2 mm, were made. Each mold was fixed to a glass plate (75×25×1 mm) by its external surface. The cement was mixed and inserted into the interior of the mold until it was completely filled. Then, the assembly was brought to an incubator at 37 ºC and relative humidity at 95%.

After 120±10 seconds from the beginning of the mixture, a 100 g Gillmore needle, with an active tip 2 mm in diameter, was placed vertically over the horizontal surface of each sample. The positioning of the Gillmore needle over the material was repeated at frequent intervals until it made no new marks in the cement being tested.

Setting time was considered to be the elapsed time between the beginning of the mixture and the moment at which marks from the Gillmore needle were no longer visible in the surface of the cement.

Radiopacity

Five teflon molds of 1.5 mm thickness, each with four perforations of 4 mm in internal diameter, were made and placed on a glass plate covered with cellophane paper.

Each perforation was filled with each group, and another glass plate wrapped in cellophane paper was placed and pressed over them. Then any excess cement was removed. The assembly was then transferred into an incubator, at 37 ºC and 95% relative humidity, and kept for a period corresponding to three times the setting time.

An acrylic plate (1.3×4.5×2 mm) was placed on this mold in order to stabilize a 99% aluminum stepwedge (made of 1100 alloy), with thickness varying from 1 to 10 mm, in uniform steps of 1 mm.

Radiographic images were obtained using a Spectro 70X (Dabi Atlante, Ribeirão Preto, Brazil) X-ray device, set to 65 kVp, 10mA, object-focus distance of 400 mm and exposure time of 0.2 s. The imaging plates, sensitized after taking the radiograph, were scanned (Digora™ Scanner) and analyzed using the *Digora for Windows 5.1* software.

Flow Test

A volume of 0.5 mL of mixed cement was placed in the center of a glass plate (10×10×3 mm), wrapped in cellophane paper. After 180±5 seconds from the start of mixing, the pliable material was placed on another glass plate also wrapped in cellophane, and an additional amount of 100 N was added carefully to the center of the material. The additional weight was removed 10 minutes after the start of the mixing and the larger and smaller diameters of the disk, obtained from the flow of the material, were measured using a digital caliper (Digimess, Shiko Precision Gaging Ltda., China).

Two conditions were necessary to validate the trial: the difference between the larger and smaller diameters could not be greater than 1 mm; and, the disk had to be uniformly circular. If not, the test was repeated.

Solubility

Circular Teflon molds, with 1.5 mm thickness and 7.75 mm internal diameter, were made and filled with the cement mixtures, and an nylon thread was included in the mass of cement. The assembly was kept in an incubator (37 °C and 95% relative humidity) for a period corresponding to three times the setting time of the cement.

After this time, the samples were removed from the mold and weighed using a precision scale (MLW, Bonn, Germany) to obtain the initial weight. Next, the samples were suspended by the nylon thread and brought to containers with 50 mL of distilled, deionized water. The containers were sealed and kept in an incubator for 7 days. After this period the samples were taken from the containers, rinsed with distilled, deionized water, dried with absorbent paper, and placed in a dehumidifier for 24 h. The samples were weighed again to obtain the final weight. The weight loss of each sample was expressed as a percentage of the original mass of the material. This was taken to be the solubility of the tested material.

Dimensional Stability

Cylindrical Teflon molds, made to obtain cylindrical specimens 12 mm high×6 mm diameter, were placed on a glass plate wrapped in cellophane paper and held in place with utility wax. The molds were over-filled with the cement and then a microscope slide, also wrapped in cellophane paper, was placed over the upper end using light pressure. The assembly was held in this position using a "C"-shaped clip. Five minutes after the start of the mixing, the assembly was brought to an incubator (37ºC and 95% relative humidity) where it was kept for a period corresponding to three times the setting time. After this period, the specimens were removed from the molds and their surfaces were ground flat using sandpaper and a scalpel blade. The initial lengths of the specimens were measured using a digital caliper; they were then placed in 10 mL plastic containers, containing 7.5 mL of deionized water, and kept in an incubator for 30 days. The samples were then removed from the containers, dried with highly absorbent paper, and their lengths were again measured, obtaining the final length of the samples.

Dimensional stability was calculated using the following formula:

$$(C^{30} - C) \div C \times 100$$

Where:
C = initial length of the specimen;
C^{30} = length of the specimen after 30 days of storage under experimental conditions.

Statistical Analysis

Five specimens from each group were tested, and their means were compared statistically. Initially, the data were submitted to preliminary testing using the GMC 8.1 software (USP, Ribeirão Preto, Brazil), which revealed a normal distribution curve. Then, parametric statistical testing (one-way ANOVA and post hoc Tukey-Kramer test) was possible, with significance level at 5% (GraphPad Software Inc., San Diego, USA).

Results

Determination of powder/liquid proportions

Group SI30 showed a lower amount of resin than the other groups (Table 1).

Table 1. Powder/Resin proportions, in grams, of the control and experimental groups.

	Resin	Control Group	Experimental Groups			
		S	SI5	SI7	SI10	SI30
		Pure Sealer®	5% iodoform	7% iodoform	10% iodoform	30% iodoform
	0.12	0.22	0.26	0.26	0.25	0.18
	0.12	0.27	0.27	0.24	0.26	0.17
	0.12	0.26	0.24	0.25	0.26	0.19
	0.12	0.26	0.26	0.27	0.23	0.19
	0.12	0.27	0.26	0.26	0.25	0.18
X±DP		0.26±0.02[a]	0.26±0.01[a]	0.26±0.01[a]	0.25±0.01[a]	0.18±0.01[b]

Values ± standard deviation.

Setting Time

ANSI/ADA (2000) demands that setting time must have a variation of no more than 10% of the time claimed by the manufacturer. The time claimed by the manufacturer of Sealer 26® cement is 12 hours, at body temperature. According to this, none of the groups comply with the norm. Statistical analysis showed the setting time for Group SI30 (4891.40±765.81 min) was significantly greater than for the other groups (P<0.001) (Table 2).

Radiopacity

All groups showed radiopacity greater than 3mm of aluminum, as recommended by ANSI/ADA Specification 57. Statistical analysis showed similarity among the groups tested (P>0.05) (Table 2).

Flow

ANSI/ADA (2000) demands that the cement have a minimum value of 20 mm in diameter; all groups showed flow within the standard demanded. Statistical analysis showed that the flow for Group SI30 (31.90±1.31 mm) was significantly greater than for the other groups tested (P<0.001) (Table 2).

Solubility

Root canal cements must not exceed 3% of the mass when the solubility of the material is tested (ANSI/ADA 2000). Except for Group SI30 (1.18±0.53%), all groups showed higher values than that recommended by ANSI/ADA standards (P<0.01) (Table 2).

Dimensional Stability

According to the ANSI/ADA (2000) standard, the cement must not have dimensions exceeding 1% contraction or 0.1% expansion. As such, none of the groups comply with Specification 57. Statistical analysis revealed similarity among Groups S (1.00±0.62%), SI5 (0.88±0.38%), and SI30 (1.96±0.69%), which were significantly lower than Groups SI7 (4.75±1.94%) and SI10 (5.57±1.60%) (P<0.05) (Table 2).

Discussion

Dental clinicians and researchers suggest the addition of some materials to the endodontic cement with the aim to maximize its properties (13,14). However, this association might be better assessed in order to elucidate its effects on the chemical reaction and on the physicochemical properties.

In this study, the high values for setting time for Sealer 26® conflict with ANSI/ADA Specification 57, which demands that a endodontic cement must not have a setting time greater than 10% of that claimed by the manufacturer. However, Garrido et al. (10) showed that Sealer 26® does comply with ADA norms for setting time. The greater setting time found for the group with 30% iodoform may be explained by the lower amount of cement powder present in the final mixture and, therefore, a lower amount of hexamethylenetetramine, a polymerization reaction accelerator in the Sealer 26®. Setting time is the time necessary for the cement to attain its defined properties and to show the stable behavior of the product, this depends on its constituent components, the size of the particles, ambient temperature and relative humidity (18). There is no standard stipulated for the setting time of cements, but clinical convenience demands that it must be long enough to manipulation and to place and adjust the filler material, if necessary (19). However, it must be as short as possible, owing to the difficulty of maintaining no moisture in the empty, prepared canal (18).

In this study, the test for radiopacity shows no statistical difference between the radiopacity of the Sealer 26® cement and the cement mixed with 5%, 7%, 10% and 30% iodoform. All groups were above 3 mm radiopacity and do comply with ADA Specification 57, which demonstrates good radiopacity in this cement independent of the addition of radiopaque agents. However, some studies show that Sealer 26® cement, in spite of its good physical, chemical and biological properties (5-10), has lower radiopacity when compared to other cements (12,20). The degree of radiopacity is essential to the control of the filling, because it shows the quality of the filling performed (21). According to the Specification 57, root canal cements should be at least 2mm Al more radiopaque than bone or dentin. Although the norms only

Table 2. Physicochemical properties of the control and experimental groups.

	Control Group	Experimental Groups			
	S	SI5	SI7	SI10	SI30
	Pure Sealer26®	5% iodoform	7% iodoform	10% iodoform	30% iodoform
Setting Time (min)	936.60±5.81[a]	1065.20±27.27[a]	1025.80±29.09[a]	1147.40±56.98[a]	4891.40±765.81[b]
Radiopacity (mm)	6.32±0.50[a]	6.41±0.89[a]	6.57±0.86[a]	6.48±0.49[a]	6.99±1.40[a]
Flow (mm)	25.67±1.20[a]	24.29±2.49[a]	24.28±2.08[a]	23.24±1.57[a]	31.90±1.31[b]
Solubility (%)	**3.39±0.32[a]**	**4.35±0.45[a]**	**3.64±1.31[a]**	**4.48±1.13[a]**	1.18±0.53[b]
Dimensional Stability (%)	**1.00±0.62[a]**	**0.88±0.38[a]**	**4.75±1.94[b]**	**5.57±1.60[b]**	**1.96±0.69[a]**

Values and standard deviation. Values followed by different superscripted letters in each line differ significantly. Values in bold type do not meet the standards demanded by ANSI/ADA (2000).

establish a lower limit for this property, it must be realized that the extreme contrast in a material may lead to a false impression of a dense and homogeneous filling (3).

The results of the flow test, similar to other studies, showed that the Sealer 26® cement complies with the standards set by ANSI/ADA (7,9). This is also the case with the other experimental groups. The increase of the flow of the group enhanced with 30% iodoform may be related to the smaller particle size of the radiopacifier agent, since the smaller the particle, the greater the flow capacity of the cement (9).

For the solubility test, with the exception of Group SI30 the other groups studied showed solubility values higher than recommended by ANSI/ADA, which sets the standard that the solubility of a material must not exceed 3% of its mass. The control group showed higher results than those found in other studies (5); and, the addition of 30% iodoform to the Sealer 26® cement reduced significantly the solubility. This suggests the formation of an insoluble compound, from the addition of this material. The high solubility of endodontic cements is undesirable because their dissolution may cause the release of materials that could irritate the periapical tissues and could permit the formation of gaps between the root canal and the filler material. This would result in an increase of leakage over time (22).

For the test of dimensional stability, all groups studied showed higher values than the ANSI/ADA (2000) specifications, which recommend that the cement must not exceed 1% contraction or 0.1% expansion. These results are similar to Carvalho-Júnior et al. (5) who observed an expansion of 3.26% of Sealer 26®. The addition of 7% and 10% iodoform to Sealer 26® produced an increase in dimensional stability. An increase in this property is also observed with the addition of calcium hydroxide to the resinous cement AH Plus (13). This dimensional alteration may be explained by the sorption of water by the resin after its polymerization (23), which may be increased with the addition of material in the cement mixture. However, the lower solubility, from the formation of an insoluble compound with the addition of 30% iodoform, may contribute to the non-increase of dimensional stability in this group.

Despite the importance of the physicochemical properties of cement is also necessary to evaluate the biological properties for a secure recommendation of cements modification.

Conclusion

The authors conclude that the addition of iodoform to the Sealer 26® cement did not alter the radiopacity property. However, it reduces the solubility and increases the values for setting time, flow, and dimensional stability.

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