Rafael Verardino de CAMARGO^(a) Yara Terezinha Corrêa SILVA-SOUSA^(b) Rodrigo Presotto Ferreira da ROSA^(c) Jardel Francisco MAZZI-CHAVES^(a) Fabiane Carneiro LOPES^(a) Liviu STEIER^(c) Manoel Damião SOUSA-NETO^(a)

(a)Universidade de São Paulo – USP, School of Dentistry of Ribeirão Preto, Department of Restorative Dentistry, Ribeirão Preto, SP, Brazil.

^(b)Universidade de Ribeirão Preto – Unaerp, Faculty of Dentistry, Ribeirão Preto, SP, Brazil.

^(e)The University of Warwick, Warwick Medical School, Coventry, United Kingdom.

Declaration of Interest: The authors certify that they have no commercial or associative interest that represents a conflict of interest in connection with the manuscript.

Corresponding Author: Manoel Damião de Sousa Neto E-mail: sousanet@forp.usp.br

https://doi.org/10.1590/1807-3107BOR-2017.vol31.0072

Submitted: Feb 23, 2017 Accepted for publication: May 29, 2017 Last revision: June 09, 2017



Evaluation of the physicochemical properties of silicone- and epoxy resin-based root canal sealers

Abstract: To assess the physicochemical properties of AH Plus, GuttaFlow 2, GuttaFlow BioSeal, and MM Seal, five samples of each root canal sealer were evaluated to determine their setting time (ST), dimensional change (DC), solubility (SL), flow (FL), and radiopacity (RD) according to American National Standards Institute/American Dental Association (ANSI/ADA) Specification 57. The distilled and deionized water obtained from the SL test were subjected to atomic absorption spectrometry to observe the presence of Ca²⁺, K⁺, and Na⁺ ions. Statistical analysis was performed by using one-way ANOVA and Tukey-Kramer tests (p < 0.05). The following results were obtained: ST (min) (AH Plus 463.6 ± 13.22; GuttaFlow 2 24.35 ± 2.78; GuttaFlow Bioseal 17.4 ± 0.55; MM Seal 47.60 ± 4.39), DC (%) (AH Plus 0.06 ± 0.12; GuttaFlow 2 - 26.06 ± 1.24; GuttaFlow Bioseal 2.10 ± 1.47; MM Seal 8.47 ± 2.41), SL (%) (AH Plus 0.41 ± 0.21; GuttaFlow 2 5.13 ± 4.11; GuttaFlow Bioseal 3.03 ± 1.05; MM Seal 0.94 ± 0.17), FL (mm) (AH Plus 36.42 ± 0.40; GuttaFlow 2 36.44 ± 0.05; GuttaFlow Bioseal 35.4 ± 0.03; MM Seal 52.75 ± 0.60), and RD (mmAl) (AH Plus 7.52 ± 1.59; GuttaFlow 2 6.85 ± 0.14; GuttaFlow Bioseal 7.02 ± 0.18; MM Seal 3.32 ± 0.90). ST, DC, SL, FL, and RD showed statistical differences among the root canal sealers (p < 0.05). As AH Plus showed the lowest DC and SL values (p < 0.05), the findings indicate that this sample is the only sealer conforming to ANSI/ADA standards.

Keywords: Silicones; Epoxy Resins; Root Canal Filling Materials

Introduction

The goal of endodontic treatment is to promote proper cleaning, shaping, and obturation of the root canal system (RCS); these are interdependent procedures that must be carefully performed so that successful treatment can be achieved¹. Three-dimensional sealing is necessary in the filling of root canals to prevent bacterial microleakage², thereby avoiding possible reinfection, and create a favorable environment for the repair of periapical tissues.^{1,2,3}

Although gutta-percha combined with the sealer AH Plus remain the most widely used materials in the filling procedure,^{1,3,4,5,6,7,8,9} numerous techniques and materials with different physicochemical and biological properties have also been developed.^{2,3,8,9,10}

Root canal sealers can be classified according to their composition as zinc oxide and eugenol-based sealers; sealers containing calcium hydroxide; glass

ionomer, epoxy resin, and methacrylate resin-based sealers; or bioceramic and silicone-based sealers^{1, 3, 7-9}.

As an epoxy resin-based sealer, AH Plus (Dentsply DeTrey, Konstanz, Germany) presents low solubility (SL) and disintegration, adequate radiopacity (RD),^{1,3,4,5,7,8,9} high bonding strength to root dentin,^{1,8,11,12} adequate expansion,^{4,5} antimicrobial activity, and other desirable biological properties.^{6,13,14}

MM-Seal (Micro-Mega, Besançon, France), a similar epoxy resin-based polymer, is composed of ethylene glycol salicylate, calcium phosphate, bismuth subcarbonate, and oxide components, thus presenting satisfactory physical and chemical properties of flow (FL) into lateral canals, RD, biocompatibility, and working time. Bodrumlu et al.,¹⁵ Bodrumlu et al.,⁵ and Poggio et al.¹⁶ reported that this root canal sealer has a bond strength comparable with that of AH Plus. However, no studies in the current literature provide an evaluation of its physicochemical properties.

In 2012, GuttaFlow 2 (Coltene/Whaledent, Langenau, Germany) was launched in the market to replace the sealer GuttaFlow. This sealer was formulated with polydimethylsiloxane, gutta-percha particulates (smaller than 30 μ m), and nano silver particles.^{14,17,18} GuttaFlow 2 is supplied in pre-mixed syringes or capsules to improve the consistency of the mixture and facilitate its use; according to its manufacturer, this sealer has an SL close to zero.

GuttaFlow Bioseal sealer (Coltene/Whaledent) was subsequently introduced to provide tissue repair along with obturation. It has the same formulation as the GuttaFlow sealer but also includes calcium silicate, which, upon contact with biological tissues, releases natural repair constituents and aids in the regeneration of periapical tissues.^{17,18,19}

To analyze the physicochemical properties of root canal sealers, the American National Standards Institute/American Dental Association (ANSI/ADA) approved Specification 57,²⁰ which proposed the carrying out FL, film thickness, setting time (ST), RD, ST, and dimensional change (DC) tests on dental sealers. In 2007, Carvalho-Junior et al.,⁴ considering the standards determined by Specification 57, suggested reducing the volume of the sealer needed for the SL and dimensional alteration tests to 80%. They also suggested complementary evaluation of the resulting liquid by atomic absorption spectrometry to analyze the ions released in the ST test.⁴

As the development of new materials, such as silicone-based sealers and their various compositions, offers the prospect of improving the quality of the resulting fillings, the objective of the present work is to evaluate the physicochemical properties of GuttaFlow 2 and GuttaFlow Bioseal (silicone-based sealers) as well as MM Seal and AH Plus (epoxy resinbased sealers) according to ANSI/ADA Specification 57 with the modifications proposed by Carvalho-Júnior et al.⁴ Here the ST, DC, SL, FL, and RD of the sealers were studied

Methodology

To accomplish this study, the following endodontic sealers were used: GuttaFlow 2, GuttaFlow Bioseal, MM Seal, and AH Plus. The tested materials were manipulated according to the manufacturer's instructions. The environment in which the experiments were performed featured a temperature of $23 \pm 2^{\circ}$ C and relative air humidity (RH) of 95% $\pm 5\%$, as recommended by ANSI/ADA Specification 57. The methodology used was proposed by the ANSI/ ADA and followed by several authors, including Resende et al.,²¹ Flores et al.,³ and Borges et al.⁷ Detailed information (material, manufacturer and composition) of the tested sealers is shown in Table 1.

Setting time

Five plaster of Paris cast rings with an internal diameter of 10 mm and a thickness of 2 mm were prepared. The external borders of the molds were fixed with wax on a glass plate. The sealers were manipulated according to the manufacturer's instructions, and the molds were filled with the material and transferred to a chamber with 95% RH and temperature of 37° C. About 150 ± 10 s after the start of mixing, a Gilmore-type needle with a mass of 100 ± 0.5 g and a flat end of 2.0 ± 0.1 mm in diameter was carefully lowered vertically into the horizontal surface of each sample. The needle tip was cleaned and probing was repeated until the indentations formed ceased to be visible. If the results differed by more than $\pm 5\%$, the test was repeated.

Materials	Manufacturer	Composition	
GuttaFlow 2	Coltène/Whaledent, Altstätten, Switzerland	Gutta-percha powder particles, polydimethylsiloxane, platinum catalyst, zirconium dioxide, micro-silver particles, coloring	
GuttaFlow BioSeal	Coltène/Whaledent, Altstätten, Switzerland	Gutta-percha powder particles, polydimethylsiloxane, platinum catalyst, zirconium dioxide, calcium salicylate, nano-silver particles, coloring, bioactive glass ceramic	
MM Seal	Micro-Mega, Besançon, France	Epoxy resin, ethylene glycol salicylate, calcium phosphate, bismuth carbonate, coloring, polyaminobenzoate, zirconium dioxide, calcium oxide	
AH Plus	Dentsply De Trey GmbH, Konstanz, Germany	Epoxy resin, calcium tungstate, zirconium dioxide, aerosil, iron oxide, adamantane amine, N,N'-dibenzyl-5-oxa-non andiamine-1,9, coloring, TCD-Diamine, silicone oil	

 Table 1. Composition of the root canal sealers and their manufacturers.

Dimensional change

Five Teflon (polytetrafluroethylene, DuPont, HABIA, Knivsta, Sweden) molds prepared for the production of 3.58 mm-high cylindrical test bodies measuring 3 mm in diameter were placed on a glass plate wrapped with a fine cellophane sheet. The molds were filled with a slight excess of freshly mixed sealer, and a microscope slide, also wrapped in cellophane, was pressed onto the upper surface of the mold. The assembled group was kept firmly joined by a C-shaped clamp, transferred to an incubator (37°C, 95% RH), and left to stand for a period corresponding to three times the ST. Afterward, the flat end of the molds containing the samples were ground with 600 grit wet sandpaper. The samples were removed from the mold, measured with a digital caliper, stored in a 50 mL vessel containing 2.24 mL of deionized distilled water, and kept in an incubator (37°C, 95% RH) for 30 days. The sample was then removed from the container, blotted dry with absorbent paper, and measured again for length. The percentage of the dimensional alterations was calculated using the formula: $[(L_{30}-L) / L] \times 100$ where L_{30} is the length of the sample after 30 days of storage and L is the initial length of the sample.

Solubility

For the SL tests, 10 circular 1.5 mm-thick Teflon molds measuring 7.75 mm in inner diameter were filled with freshly mixed sealer. Each mold was supported by a larger glass plate and covered with a cellophane sheet. An impermeable nylon thread was placed inside the material, and another glass plate, also covered with cellophane film, was positioned on the mold and pressed manually in such a way that the plates touched the entire mold in a uniform manner. The assembly was placed in an incubator (37°C, 95% RH) and left to stand for a period corresponding to three times the ST. As soon as the samples were removed from the mold, they were weighed three times each (HM-200, A&D Engineering, Inc., Bradford, MA, USA), and the mean reading was recorded. The samples were suspended by nylon thread and placed two-by-two inside a plastic vessel with a wide opening containing 7.5 mL of deionized distilled water, taking care to avoid any contact between them and the inner surface of the container. The containers were sealed and left for 7 days in an incubator (37°C, 95% RH). Afterward, the samples were removed from the containers, rinsed with deionized distilled water, blotted dry with absorbent paper, and placed in a dehumidifier for 24 h. They were

then weighed again. The weight loss of each sample (initial mass minus final mass) was expressed as a percentage of the original mass ($m\% = m_i - m_f$) and taken as the SL of the sealer.

A volume of 7.5 mL of distilled water was taken from each sample and poured into a clean and dry porcelain crucible. Each crucible was put into a muffle furnace and burned at 550°C. The resulting ash was dissolved in 10 mL of concentrated nitric acid (Merck KGaA, Darmstadt, Germany) using a glass stick. Thereafter, the samples were poured into 50 mL volumetric flasks, and the volume was made up with ultrapure deionized water (MilliQ, Millipore, Billerica, MA, USA). The solutions were sprayed into an atomic absorption spectrophotometer (Perkin Elmer, Uberlingen, Germany) to verify the presence of Ca²⁺, Na⁺, and K⁺ ions. The arithmetic mean of three replicates for each specimen was recorded and considered as the result, expressed as mg/L.

Flow

For the FL tests, 0.5 mL of the sealer was manipulated. About 3 minutes after the start of mixing, the sealer was placed in the center of a glass plate (40 mm × 40 mm × 3 mm) with the aid of a graduated syringe. Then, a second glass plate weighing 120 g was placed onto the sample. After 10 minutes, the largest and smallest diameters of the samples were measured using a digital caliper. In this test, the difference between the larger and smaller diameters should not exceed 1 mm, and the sealers should be uniform.

Radiopacity

Five acrylic plates (2.2 cm × 4.5 cm × 1 mm) containing four wells measuring 1 mm in depth and 5 mm in diameter were prepared and placed over a glass plate covered by a cellophane sheet. Each well was filled with one of the sealers, following a sequence according to the ST of the material so that the samples were ready for radiographic evaluation immediately after the final setting of all materials. To avoid the formation of bubbles, the freshly mixed sealer was introduced into the wells using a syringe. Another glass plate covered with cellophane was placed on top of the samples until complete setting,

and any excess sealer was removed. Each plate was kept in an incubator (37°C, 95% RH) for a period corresponding to three times the ST.

At the time of radiographic exposure, each of the acrylic plates containing the sealers was positioned alongside another acrylic plate (1.3 cm × 4.5 cm × 1 mm) containing an aluminum step wedge made of 1100 alloy with the thickness varying from 1 mm to 10 mm in uniform steps of 1 mm each (Margraf Dental MFG Inc., Jenkintown, PA, USA). This set of acrylic plates was placed in front of a phosphor plate next to the aluminum step wedge, and a digital radiograph was taken (DigoraTM system; Soredex Orion Corporation, Helsinki, Finland). Radiographic images were obtained using a Spectro 70X X-ray machine (Dabi Atlante, Ribeirão Preto, SP, Brazil) at 70 kVp and 8 mA. The object-to-focus distance was 30 cm, and the exposure time was 0.2 s. Exposed imaging plates of the test samples were immediately scanned after exposure (DigoraTM Scanner) and analyzed using DigoraTM for Windows 5.1 software.

Digora[™] software provides the radiographic density determination (densitometric analysis) or, in other words, the RD of a given material through its gray levels (mmAl). Thus, a 2 mm² area (44.5 × 44.5 px²) was standardized and used for each specimen in the radiographic images of the sealers.

Statistical analysis

Five specimens from each group were tested and the means were statistically compared with the aid of SPSS Statistics 17.0 software (LEAD Technologies, Inc., Chicago, IL, USA). The Kolmogorov–Smirnov test showed that the results were consistent with a normal distribution curve; thus, parametric statistical analysis was possible (one-way ANOVA and post hoc Tukey–Kramer test), and the null hypothesis was set as 5%.

Results

Table 2 shows the analysis results of the physicalchemical properties of the root canal sealers.

Setting time

Among the sealers tested, AH Plus presented the highest mean values, and the difference in results

between this sealer and those of the other sealers was statistically significant (p < 0.05) (Table 2). According to ANSI/ADA Specification 57, all root canal filling materials should not have an ST greater than 10% of the time determined by the manufacturer. Therefore, GuttaFlow Bioseal, MM Seal, and AH Plus conform to the recommended specifications.

Dimensional change

Among the sealers tested, GuttaFlow 2, GuttaFlow Bioseal, and MM Seal sealers showed the highest mean values, and statistically significant differences were observed among groups. AH Plus presented the lowest mean value, and this difference was statistically significant when compared with the values of the other sealers (p < 0.05) (Table 2). ANSI/ADA Specification 57 recommends that sealers should not exceed 1% contraction or 0.1% expansion.

Solubility

GuttaFlow 2 and GuttaFlow Bioseal presented the highest mean values and standard deviations in the SL test, and the results of these sealers were statistically similar to each other and statistically different when compared with those of AH Plus, which presented the lowest averages (p < 0.05) (Table 2). ANSI/ADA Specification 57 stipulates that an ideal endodontic sealer should not lose more than 3% of its mass when its SL is tested.

The amounts of metal ions released into the liquid resulting from immersion of the SL test samples are listed in Table 3. The data in Table 3 show that GuttaFlow 2 and GuttaFlow Bioseal present higher calcium release rates than either MM Seal or and AH Plus and that GuttaFlow Bioseal is the only sealer with a high sodium release rate. All of the sealers showed low rates of potassium release.

Flow

All of the sealers presented similarly intermediate values, with no statistically significant difference among them (p > 0.05) (Table 2). ANSI/ADA Specification 57 states that sealers should not have a difference greater than 1 mm between the largest and smallest diameters of the flowed material.

Radiopacity

The radiographic densities of the sealers were obtained in gray scale. MM Seal showed the lowest mean values and standard deviations, and differences between the results of this and other groups were statistically different (p < 0.05) (Table 2). According to ANSI/ADA Specification 57, all root canal filling materials must have an RD greater than or equal to 3 mmAl. All of the sealers presented an RD greater than 3 mmAl.

Table 2. Physicochemical properties of GuttaFlow 2, GuttaFlow Bioseal, MM Seal, and AH Plus (mean ± SD).

Variable	GuttaFlow 2	GuttaFlow BioSeal	MM Seal	AH Plus
Setting time (min)	$24.35\pm2.78^{\circ}$	$17.4\pm0.55^{\rm b}$	$47.60\pm4.39^{\circ}$	$463.60\pm13.22^{\rm d}$
Dimensional change (%)	$-26.06 \pm 1.24^{\circ}$	$2.10\pm1.47^{\rm b}$	$8.47 \pm 2.41^{\circ}$	$0.06\pm0.12^{\rm d}$
Solubility (%)	$5.13 \pm 4.11^{\circ}$	$3.03 \pm 1.05^{\circ}$	$0.94\pm0.17^{\rm b}$	$0.41 \pm 0.21^{\circ}$
Flow (mm)	$36.44\pm0.05^{\circ}$	$35.4\pm0.03^{\circ}$	$52.75\pm0.60^{\rm b}$	$36.42\pm0.40^{\circ}$
Radiopacity (mmAl)	$6.85 \pm 0.14^{\circ}$	$7.02 \pm 0.18^{\circ}$	$3.32\pm0.90^{\rm b}$	7.52 ± 1.59°

Values followed by superscript lowercase letters indicate statistical differences between columns (p > 0.05).

Table 3. Metal solubility (mg/L) c	f root canal sealers determined by	atomic absorption spectrometry.
------------------------------------	------------------------------------	---------------------------------

Metal ions	GuttaFlow 2	GuttaFlow BioSeal	MM Seal	AH Plus
Ca ²⁺	1.97 ± 0.71	2.23 ± 0.92	1.72 ± 2.73	< 1.00
K+	0.87 ± 0.50	0.90 ± 0.15	0.29 ± 0.03	0.14 ± 0.04
Na+	1.46 ± 0.49	732.97 ± 124.32	0.75 ± 0.13	0.58 ± 0.21

Discussion

In 1983, the ADA proposed a set of standards and tests known as Specification 57 to evaluate the physicochemical properties of endodontic filling materials. This specification determines the following tests for physicochemical assessment: FL, film thickness, ST, RD, SL, and dimensional stability (ADA, 1984). In 2000, ANSI/ADA Specification 57 changed the SL and disintegration designation, which was established in 1984, to SL.²⁰

The methodology used in this study followed ANSI/ADA Specification 57 with the modifications proposed by Carvalho-Junior et al.,⁴ followed by Resende et al.,²¹ Marin-Bauza et al.,⁶ and Flores et al.³ These modifications suggested reduction of the volume of the sealer needed for the SL and dimensional stability tests by 80% to reduce the consumption of the test material without changing results. The authors further suggested analysis of the resulting liquid in the SL tests by atomic absorption spectrometry to evaluate ions released by the sealing materials.^{34,6,21}

The silicon-based sealers GuttaFlow 2 (24'35" \pm 2'78") and GuttaFlow BioSeal (17'40" \pm 0'55") presented the lowest STs. This finding can be attributed to the presence of polydimethylsiloxane polymers in the sealers, which promote the polymerization reaction between the polydimethylsiloxane, silicone oils, and paraffin catalyzed by platinum.^{14,17,18,22} These silicon-based sealers also include gutta-percha, silver nanoparticles, and calcium silicate in their composition, all of which work as fillers and remain in the polymer cavities instead of participating in the polymerization, which results in significantly reduced STs.^{14,17,18,22} The results found in this study contrast those of Gandolfi et al.,¹⁸ who showed higher STs for these two sealers.¹⁸

AH Plus ($464' \pm 13'22''$) and MM Seal ($48' \pm 4'39''$) presented the highest STs, similar to findings reported in the literature.^{1,3,5,7,8,9,13,14,15,16,21,23} This result is likely due to the occurrence of slow polymerization between the epoxy resin amines and high-molecular weight molecules. Conversion of monomers into polymers occurs gradually.²³ These two sealers also include aliphatic and aromatic amines in their formulation, which are responsible for their longer STs.^{1,5,7,8,9,13,14,15,16,21,23}

The sealers tested in the present study conformed to the standards proposed by ANSI/ADA since their STs did not exceed 10% of those values determined by their respective manufacturers, *i.e.*, 25–30 minutes for GuttaFlow 2, 12–16 minutes for GuttaFlow Bioseal, 45 minutes for MM Seal, and 480 minutes for AH Plus sealer. The ST is an initial test of the sealers and could serve as a parameter for other tests; results may vary according to the components and particle size of the tested material, ambient temperature, and RH.^{318,24,25,26,27}

The DC test showed higher values for GutaFlow 2 (-26.06% \pm 1.24%) and GutaFlow Bioseal (2.10% \pm 1.47%) in comparison with those for MM Seal (8.47% \pm 2.41%) and AH Plus (0.06% \pm 0.12%), possibly due to the polymer reaction between bisphenol A and F resins, which promote a closer and more rigid structure through hydrogen bonds and allow the adsorption of water after polymerization.^{1,3,5,6,7,8,9,13,14,15,16,21,23}

GuttaFlow 2 showed a significant loss of sample mass, likely because of the presence of gutta-percha and silver nanoparticles, which confer greater stability to the polymer matrix due to attractive forces and cross-linking between molecules. Although GuttaFlow Bioseal also presents these compounds in its formulation, addition of calcium silicate to this cement, which features hygroscopic properties, promotes water accumulation and the consequent mass gain.^{28,29} Thus, considering that no material should contract or expand by more than 1% or 0.1%, respectively, only AH Plus conforms to the standards proposed by ANSI/ ADA Specification 57.

In this study, the highest SL values were observed for GuttaFlow 2 (5.13 ± 4.11) and GuttaFlow BioSeal (3.03 ± 1.05); these values exceed the 3% mass loss determined by ANSI/ADA. The lowest averages were found for MM Seal (0.94 ± 0.17) and AH Plus (0.41 ± 0.21) and are attributed to the fact that silicone-based sealers include gutta-percha in their composition, which could increase the size of the polymer cavities of the polydimethylsiloxane and oils. Larger pore cavities yield a more open molecular structure and allow greater water absorption. The hygroscopic capacity of the calcium silicate present in GuttaFlow Bioseal may result in the greatest accumulation of water between the chains of this polymer.^{19,30}

The results of AH Plus and MM Seal are in agreement with those of Bodrumlu et al.,¹⁵ Resende et al.,²¹ Bodrumlu et al.,⁵ Marin-Bauza et al.,⁶ Poggio et al.,¹⁶ Flores et al.,³ Sonntag et al.,³¹ and Prüllage et al.,²⁷ which suggests that their low SL may be related to their composition and polymerization reactions. When the two pastes are mixed, the diepoxy and polyamine compounds react, forming a covalent reaction. Amine groups promote changes in the ST, density, and morphology of the epoxy resins. Each amine group can react with an epoxy group, resulting in the formation of a strong and compact cross-linked polymer, which promotes low levels of SL.³⁷

Complementary analysis of the solutions by means of atomic absorption spectrometry showed the significant release of calcium ions from all of the evaluated sealers, as well as the release of sodium ions from GuttaFlow Bioseal (732.97 \pm 124.32). The high rate of sodium release from this sealer is due to the addition of bioactive glass ceramics in its composition. The material is mainly composed of sodium oxide, which stimulates the formation of mineralized tissue.^{32,33,34,35,36} Calcium and sodium ion release increases the pH of the dental environment and prohibits bacterial viability.³⁶

In the FL test, all of the sealers presented a diameter greater than 20 mm as determined by ANSI/ADA. The flowabilities of GuttaFlow 2 (36.44 ± 0.05 mm) and GuttaFlow BioSeal (35.40 ± 0.03 mm) can be attributed to the reaction between the polydimethylsiloxane, silicone oil, and paraffin they contain, as well as the pressure exerted by the glass plate during the test, once these sealers have thixotropic capacity under

compression, which allows the fluidity and greater penetrability of these sealers. $^{3,14\,17\,18\,37,38}$

The FL test results of MM Seal (52.75 ± 0.60 mm) and AH Plus (36.42 ± 0.40 mm) can be attributed to the presence of thickening agents and polymerization activators, including ethylene glycol salicylate, aerosil, the adamantylamine, and *N*,*N*-dibenzyl-5-oxanonane-diamine-1,9 in their composition, which promotes the thixotropy of the internal structure of the sealers during the polymerization reaction and increases their fluidity.^{1,3,5,6,7,8,9,13,14,15,16,21}

In the RD test, all of the sealers presented radiographic densities higher than the recommended 3 mmAI determined by ANSI/ADA Specification 57.^{3,4} ^{6 21,39} The RDs of GutaFlow 2 (6.85 ± 0.14 mmAl) and GutaFlow Bioseal (7.02 ± 0.18 mmAl) are related to the presence of radiopacifiers such zirconium dioxide and nano silver particles in their formulation,^{3,14,718} while those of MM Seal (3.32 ± 0.90 mmAl) and AH Plus (7.52 ± 1.59 mmAl) are due to the presence of zirconium oxide, iron oxide, and calcium tungstate in their composition.^{1,3,4,8,9,14,24,40} The results found in the present study are in accordance with those of other studies, which found similar radiographic densities for AH Plus^{3,4,6,21,26,27,31,39} and for GuttaFlow 2 and GuttaFlow Bioseal.^{3,14,17,18}

Conclusion

According to the methodology employed and the results obtained in this study, AH Plus can be concluded to be the only sealer that fulfills ANSI/ ADA specifications. New studies should be carried out to better interpret the physicochemical properties of endodontic sealers and provide more support to researchers and clinicians attempting to develop an ideal sealer.

References

- Albino Souza M, Dalla Lana D, Gabrielli E, Barbosa Ribeiro M, Miyagaki DC, Cecchin D. Effectiveness of final decontamination protocols against Enterococcus faecalis and its influence on bond strength of filling material to root canal dentin. Photodiagnosis Photodyn Ther. 2017;17:92-7. https://doi.org/10.1016/j.pdpdt.2016.11.004
- Silva EJ, Carvalho NK, Prado MC, Zanon M, Senna PM, Souza EM et al. Push-out bond strength of injectable pozzolan-based root canal sealer. J Endod. 2016;42(11):1656-9. https://doi.org/10.1016/j.joen.2016.08.009

- Flores DS, Rached FJ Jr, Versiani MA, Guedes DF, Sousa-Neto MD, Pécora JD. Evaluation of physicochemical properties of four root canal sealers. Int Endod J. 2011; 44(2):126-35. https://doi.org/10.1111/j.1365-2591.2010.01815.x
- Carvalho-Junior JR, Correr-Sobrinho L, Correr AB, Sinhoreti MA, Consani S, Sousa-Neto MD. Radiopacity of root filling materials using digital radiography. Int Endod J. 2007; 40(7):514-20. https://doi.org/10.1111/j.1365-2591.2007.01246.x
- Bodrumlu E, Parlak E, Bodrumlu EH. The effect of irrigation solutions on the apical sealing ability in different root canal sealers. Braz Oral Res. 2010;24(2):165-9. https://doi.org/10.1590/S1806-83242010000200007
- Marin-Bauza GA, Rached-Junior FJ, Souza-Gabriel AE, Sousa-Neto MD, Miranda CE, Silva-Sousa YT. Physicochemical properties of methacrylate resin-based root canal sealers. J Endod. 2010;36(9):1531-6. https://doi.org/10.1016/j.joen.2010.05.002
- 7. Borges RP, Sousa-Neto MD, Versiani MA, Rached-Júnior FA, De-Deus G, Miranda CE et al. Changes in the surface of four calcium silicate-containing endodontic materials and an epoxy resin-based sealer after a solubility test. Int Endod J. 2012;45(5):419-28. https://doi.org/10.1111/j.1365-2591.2011.01992.x
- De-Deus G, Belladonna FG, Silva EJ, Souza EM, Carvalhal JC, Perez Ret al. Micro-CT assessment of dentinal micro-cracks after root canal filling procedures. Int Endod J. Forthcoming 2016. https://doi.org/10.1111/iej.12706
- Kaşıkçı Bilgi I, Köseler I, Güneri P, Hülsmann M, Çalışkan MK. Efficiency and apical extrusion of debris: a comparative ex vivo study of four retreatment techniques in severely curved root canals. I Int Endod J. Forthcoming 2016. Htps://doi.org/10.1111/iej.12708
- Prati C, Gandolfi MG. Calcium silicate bioactive cements: biological perspectives and clinical applications. Dent Mater. 2015;31(4):351-70. https://doi.org/10.1016/j.dental.2015.01.004
- Martins CV, Leoni GB, Oliveira HF, Arid J, Queiroz AM, Silva LA et al. Influence of therapeutic cancer radiation on the bond strength of an epoxy- or an MTA-based sealer to root dentine. Int Endod J. 2016;49(11):1065-72. https://doi.org/10.1111/iej.12556
- Franceschini KA, Silva-Sousa YT, Lopes FC, Pereira RD, Palma-Dibb RG, de Sousa-Neto MD. Bond strength of epoxy resin-based root canal sealer to human root dentin irradiated with Er,Cr:YSGG laser. Lasers Surg Med. 2016;48(10):985-94. https://doi.org/10.1002/lsm.22496
- Arias-Moliz MT, Camilleri J. The effect of the final irrigant on the antimicrobial activity of root canal sealers. J Dent. 2016;52:30-6. https://doi.org/10.1016/j.jdent.2016.06.008
- 14. Shakya VK, Gupta P, Tikku AP, Pathak AK, Chandra A, Yadav RK et al. An invitro evaluation of antimicrobial efficacy and flow characteristics for AH Plus, MTA Fillapex, CRCS and gutta flow 2 root canal sealer. J Clin Diagn Res. 2016;10(8):ZC104-8. https://doiorg/10.7860/JCDR/2016/20885.8351.

- Bodrumlu E, Avsar A, Meydan AD, Tuloglu N. Can radiotherapy affect the apical sealing ability of resin-based root canal sealers? J Am Dent Assoc. 2009;140(3):326-30. https://doi.org/10.14219/jada.archive.2009.0162
- Poggio C, Arciola CR, Dagna A, Colombo M, Bianchi S, Visai L. Solubility of root canal sealers: a comparative study. Int J Artif Organs. 2010;33(9):676-81.
- 17. Akcay M, Arslan H, Durmus N, Mese M, Capar ID. Dentinal tubule penetration of AH Plus, iRoot SP, MTA fillapex, and guttaflow bioseal root canal sealers after different final irrigation procedures: a confocal microscopic study. Lasers Surg Med. 2016;48(1):70-6. https://doi.org/10.1002/lsm.22446
- Gandolfi MG, Siboni F, Prati C. Properties of a novel polysiloxane-guttapercha calcium silicate-bioglass-containing root canal sealer. Dent Mater. 2016;32(5):e113-26. https://doi.org/10.1016/j.dental.2016.03.001
- Zeigler JM, Fearon FWG, ed. Silicon-based polymer science: a comprehensive resource. Washigton, DC: American Chemical Society; 1990. (Advances in chemistry series, Vol 224).
- American National Standards, American Dental Association. ANSI/ADA Specification No. 57: endodontic sealing material. Chicago: American National Standards; 2000.
- Resende LM, Rached-Junior FJ, Versiani MA, Souza-Gabriel AE, Miranda CE, Silva-Sousa YT et al. A comparative study of physicochemical properties of AH Plus, Epiphany, and Epiphany SE root canal sealers. Int Endod J. 2009;42(9):785-93. https://doi.org/10.1111/j.1365-2591.2009.01584.x
- 22. Katsamakis S, Slot DE, Van der Sluis LW, Van der Weijden F. Histological responses of the periodontium to MTA: a systematic review. J Clin Periodontol. 2013;40(4):334-44. https://doi.org/10.1111/jcpe.12058
- Lin-Gibson S, Landis FA, Drzal PL. Combinatorial investigation of the structure-properties characterization of photopolymerized dimethacrylate networks. Biomaterials. 2006;27(9):1711-7. https://doi.org/10.1016/j.biomaterials.2005.10.040
- 24. Ørstavik D, Nordahl I, Tibballs JE. Dimensional change following setting of root canal sealer materials. Dent Mater. 2001;17(6):512-9. https://doi.org/10.1016/S0109-5641(01)00011-2
- 25. Wang J, Zuo Y, Zhao M, Jiang J, Man Y, Wu J et al. Physicochemical and biological properties of a novel injectable polyurethane system for root canal filling. Int J Nanomedicine. 2015;10:697-709. https://doi.org/10.2147/IJN.S74025
- 26. Khalil I, Naaman A, Camilleri J. Properties of Tricalcium Silicate Sealers. J Endod. 2016;42(10):1529-35. https://doi.org/10.1016/j.joen.2016.06.002
- 27. Prüllage RK, Urban K, Schäfer E, Dammaschke T. Material Properties of a Tricalcium Silicate-containing, a Mineral Trioxide Aggregate-containing, and an Epoxy Resinbased Root Canal Sealer. J Endod. 2016;42(12):1784-8. https://doi.org/10.1016/j.joen.2016.09.018

- 28. Gandolfi MG, Iacono F, Agee K, Siboni F, Tay F, Pashley DH et al. Setting time and expansion in different soaking media of experimental accelerated calciumsilicate cements and ProRoot MTA. Oral Surg Oral Med Oral Pathol Oral Radiol Endod. 2009;108(6):e39-45. https://doi.org/10.1016/j.tripleo.2009.07.039
- Chiu CK, Lee DJ, Chen H, Chow LC, Ko CC. In-situ hybridization of calcium silicate and hydroxyapatite-gelatin nanocomposites enhances physical property and in vitro osteogenesis. J Mater Sci Mater Med. 2015;26(2):92. https://doi.org/10.1007/s10856-015-5456-9
- 30. Gong W, Zeng K, Wang L, Zheng S. Poly (hydroxyether of bisphenol A)-block-polydimethylsiloxane alternating block copolymer and its nanostructured blends with epoxy resin. Polymer. 2008;49(15):3318-26. https://doi.org/10.1016/j.polymer.2008.05.032
- 31. Sonntag D, Ritter A, Burkhart A, Fischer J, Mondrzyk A, Ritter H. Experimental amine-epoxide sealer: a physicochemical study in comparison with AH Plus and EasySeal. Int Endod J. 2015;48(8):747-56. https://doi.org/10.1111/iej.12372
- 32. Kokubo T. Bioactive glass ceramics: properties and applications. Biomaterials. 1991;12(2):155-63. https://doi.org/10.1016/0142-9612(91)90194-F
- Sepulveda P, Jones JR, Hench LL. Characterization of melt-derived 45S5 and sol-gel-derived 58S bioactive glasses. J Biomed Mater Res. 2001;58(6):734-40. https://doi.org/10.1002/jbm.10026

- Montazerian M, Zanotto ED. Bioactive and inert dental glass-ceramics. J Biomed Mater Res A. 2017; 105(2):619-39. https://doi.org/10.1002/jbm.a.35923
- 35. Placek LM, Keenan TJ, Wren AW. Bioactivity of Y2O3 and CeO2 doped SiO2-SrO-Na2O glassceramics. J Biomater Appl. 2016;31(2):165-80. https://doi.org/10.1177/0885328216651392
- 36. Carvalho CN, Freire LG, Carvalho AP, Duarte MA, Bauer J, Gavini G. Ions Release and pH of Calcium Hydroxide-, Chlorhexidine- and Bioactive Glass-Based Endodontic Medicaments. Braz Dent J. 2016;27(3):325-31. https://doi.org/10.1590/0103-6440201600602
- Zielinski TM, Baumgartner JC, Marshall JG. An evaluation of Guttaflow and gutta-percha in the filling of lateral grooves and depressions. J Endod. 2008;34(3):295-8. https://doi.org/10.1016/j.joen.2007.12.004
- Zhou HM, Shen Y, Zheng W, Li L, Zheng YF, Haapasalo M. Physical properties of 5 root canal sealers. J Endod. 2013;39(10):1281-6. https://doi.org/10.1016/j.joen.2013.06.012
- 39. Tanomaru-Filho M, da Silva GF, Duarte MA, Gonçalves M, Tanomaru JM. Radiopacity evaluation of root-end filling materials by digitization of images. J Appl Oral Sci. 2008;16(6):376-9. https://doi.org/10.1590/S1678-77572008000600004
- Tanomaru-Filho M, Jorge EG, Guerreiro Tanomaru JM, Gonçalves M. Radiopacity evaluation of new root canal filling materials by digitalization of images. J Endod. 2007; 33(3):249-51. https://doi.org/10.1016/j.joen.2006.08.015