

## Evaluation of physicochemical properties of four root canal sealers

D. S. H. Flores<sup>1</sup>, F. J. A. Rached-Júnior<sup>2</sup>, M. A. Versiani<sup>1</sup>, D. F. C. Guedes<sup>1</sup>, M. D. Sousa-Neto<sup>1</sup> & J. D. Pécora<sup>1</sup>

<sup>1</sup>Department of Restorative Dentistry, Faculty of Dentistry of Ribeirão Preto, University of São Paulo (FORP-USP), Ribeirão Preto, SP, Brazil; and <sup>2</sup>Department of Endodontics, Faculty of Dentistry, University of Ribeirão Preto, Ribeirão Preto, SP, Brazil

### Abstract

**Flores DSH, Rached-Júnior FJA, Versiani MA, Guedes DFC, Sousa-Neto MD, Pécora JD.** Evaluation of physicochemical properties of four root canal sealers. *International Endodontic Journal*, **44**, 126–135, 2011.

**Aim** To assess the physicochemical properties and the surface morphology of AH Plus, GuttaFlow, RoekoSeal and Activ GP root canal sealers.

**Methodology** Five samples of each material were evaluated for setting time, dimensional alteration, solubility and radiopacity tests, according to ANSI/ADA Specification 57. A total of 50 mL of deionized distilled water from the solubility tests were used to measure the metal solubility by atomic absorption spectrometry. The morphologies of the external surface and the cross-section of the samples were analysed by means of a scanning electron microscope (SEM). Statistical analysis was performed by using one-way ANOVA and *post hoc* Tukey–Kramer tests with the null hypothesis set as 5%.

**Results** AH Plus had the longest setting time ( $580.6 \pm 3.05$  min) ( $P < 0.05$ ). Activ GP did not have a mean value on the radiopacity and solubility tests

( $1.31 \pm 0.35$  mm and  $11.8 \pm 0.43\%$ , respectively) in accordance with ANSI/ADA, being significantly different from the other materials ( $P < 0.05$ ), which had mean values for these tests in accordance with the ADA's requirements. GuttaFlow was the only sealer that conformed to the Specification 57 concerning the dimensional alteration test ( $0.44 \pm 0.16\%$ ) ( $P < 0.05$ ). The spectrometry test revealed significant  $\text{Ca}^{2+}$ ,  $\text{K}^{+}$ ,  $\text{Zn}^{2+}$  ion release from Activ GP sealer ( $32.57 \pm 5.0$ ,  $1.57 \pm 0.22$  and  $8.20 \pm 1.74$   $\mu\text{g mL}^{-1}$ , respectively). In SEM analysis, the loss of matrix was evident and the filler particles were more distinguishable in all groups.

**Conclusions** The setting time of all sealers was in accordance with ANSI/ADA's requirements. Activ GP did not fulfill ANSI/ADA's protocols regarding radiopacity, dimensional alteration and solubility. GuttaFlow was the only sealer that conformed to the Specification 57 in all tests. SEM analysis revealed that the surfaces of all sealers had micromorphological changes after the solubility test.

**Keywords:** endodontics, physicochemical properties, root canal filling, sealer.

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### Introduction

In root canal treatment, complete sealing of the root canal system after cleaning and shaping is critical to prevent oral pathogens from colonizing and re-infecting the root and periapical tissues. Although gutta-percha

is still the most commonly used root canal filling material, a number of new techniques and materials with different physicochemical properties have been developed (Ørstavik 2005, Versiani *et al.* 2006, Resende *et al.* 2009).

During canal filling procedures, the endodontic sealer performs several functions including the filling of root canal wall irregularities such as apical ramifications and deltas as well as spaces where gutta-percha was not able to adapt (Kontakiotis *et al.* 2007). Because

Correspondence: Manoel D. Sousa Neto, Rua Célia de Oliveira Meirelles 350, 14024-070 Ribeirão Preto, SP, Brazil (Tel./fax: +55-16-3603-6783; e-mail: sousanet@forp.usp.br).

the conventional filling materials do not effectively seal the root canal space, new endodontic sealers have been developed (Bouillaguet *et al.* 2008).

Although AH Plus has adequate long-term dimensional stability, its sealing ability remains controversial partly because AH Plus does not bond to gutta-percha (Ørstavik *et al.* 2001). RoekoSeal Automix (Coltène Whaledent, Langenau, Germany) is a sealer containing dimethylpolysiloxane with limited data available on its physicochemical properties (Gençoglu *et al.* 2003). Despite the promising clinical and laboratory data reported for the silicone-based sealer, RoekoSeal has been tested in various studies with contradictory results (De-Deus *et al.* 2007). GuttaFlow (Coltène Whaledent, Alstätten, Switzerland) is a polyvinylsiloxane-based sealer and was introduced with the intention of reducing the disadvantages of warm gutta-percha techniques (Eldeniz & Ørstavik 2009). It has been claimed that GuttaFlow could improve the seal because of increased flowability and the fact that the material expands slightly on setting (Bouillaguet *et al.* 2008, Eldeniz & Ørstavik 2009, Roggendorf *et al.* 2010).

Dental applications of glass ionomer sealers take advantage of their bond to dentine, fluoride release, antimicrobial activity and biocompatibility (Ørstavik 2005). However, some laboratory tests have indicated a propensity for leakage and disintegration (Carvalho-Júnior *et al.* 2003, Schäfer & Zandbiglari 2003). Recently, Activ GP Precision Obturation System (Brasseler USA, Savannah, GA, USA) was marketed as a root filling monoblock system by using conventional gutta-percha cones that are surface coated with glass ionomer fillers (Roggendorf *et al.* 2010). The manufacturer claims the product to be superior to previous glass ionomer-based systems in terms of handling characteristics, working time, radiopacity (Fransen *et al.* 2008), and seal, because of the increased flowability and the fact that the material expands slightly on setting (Kontakiotis *et al.* 2007).

Although various kinds of endodontic sealers have been proposed as innovative filling materials, the ideal root canal sealer has yet to be found (Ørstavik 1983, 2005, Ørstavik *et al.* 2001, Versiani *et al.* 2006, Resende *et al.* 2009). Therefore, the aim of this laboratory study was to compare the setting time, radiopacity, dimensional change, and solubility of silicone-based sealers (GuttaFlow and RoekoSeal), and an ionomer-based sealer (Activ GP) with a well-established epoxy resin-based sealer (AH Plus), according to ANSI/ADA (2000) standards.

## Materials and methods

Setting time, radiopacity, dimensional changes, and solubility, after setting for AH Plus (group I), GuttaFlow (group II), RoekoSeal (group III), and Activ GP (group IV) root canal sealers were measured according to ANSI/ADA (2000) standards for root canal sealing materials. The tested materials (Table 1) were manipulated according to the manufacturer's instructions. Analyses were performed by a single examiner who was blind to the experimental materials. To standardize and homogenize the amount of material, 0.3 g of sealer was dispensed onto a mixing pad and spatulated for 15 s for each tested sample. For physicochemical tests, the arithmetic mean of five replicates for each sealer was recorded and considered as the result of the test.

### Setting time

Five plaster of Paris cast rings, having an internal diameter of 10 mm and a thickness of 2 mm were prepared. The external borders of the moulds were fixed with wax on a glass plate (75 × 25 × 1 mm). The moulds were then filled with the material and transferred to a chamber with 95% relative humidity (RH) and a temperature of 37 °C. After 150 ± 10 s from the start of mixing the sealer, a Gilmore-type needle with a mass of 100 ± 0.5 g having a flat end of 2.0 ± 0.1 mm in diameter was carefully lowered vertically onto the horizontal surface of each sample. The needle tip was cleaned and the probing was repeated until indentations ceased to be visible. If the results differed by more than ±5%, the test was repeated.

### Radiopacity test

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 setting time of the material, from the longest to the shortest, so that the samples would be ready for radiographic evaluation after the final setting of all materials. In order 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 until complete setting and any excess sealer removed. Each plate was kept in an incubator (37 °C, 95% RH) for a period corresponding to three times the setting time.

**Table 1** Composition of the materials and their manufacturers

| Materials | Components                                                                                          |                                                                                                                                                                                                    | Manufacturer                                 |
|-----------|-----------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------|
| AH Plus   | <i>Component A:</i><br>Epoxy resin<br>Calcium tungstate<br>Zirconium oxide<br>Aerosil<br>Iron oxide | <i>Component B:</i><br>Adamantane amine<br><i>N,N</i> -Dibenzyl-5-oxanonane<br>TCD-Diamine<br>Calcium tungstate<br>Zirconium oxide<br>Aerosil                                                      | Dentsply De Trey GmbH,<br>Konstanz, Germany  |
| GuttaFlow | <i>Guttaflow Primer:</i><br>Heptane                                                                 | <i>GuttaFlow:</i><br>Polydimethylsiloxane<br>Gutta-percha powder<br>Zinc oxide<br>Zirconium dioxide<br>Nano-silver<br>Paraffin oil<br>Hexachloroplatinic acid<br>Silicic acid<br>Platinum catalyst | Coltène Whaledent, Alstätten,<br>Switzerland |
| RoekoSeal | <i>Component A:</i><br>Polymethylsiloxane<br>Silicone oil<br>Paraffin-base oil                      | <i>Component B:</i><br>Hexachloroplatinic acid<br>Zirconium dioxide                                                                                                                                | Coltène Whaledent, Langenau,<br>Germany      |
| Activ GP  | <i>Powder:</i><br>Barium aluminosilicate<br>Glass powder<br>Dried polyacrylic acid                  | <i>Liquid:</i><br>Polyacrylic acid<br>Tartaric acid                                                                                                                                                |                                              |

Each one of the acrylic plates containing the sealers was positioned, at the time of the radiographic exposure, alongside to another acrylic plate (1.3 cm × 4.5 cm × 1 mm), containing an aluminium stepwedge, made of 1100 alloy, with the thickness varying from 1 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 this phosphor plate, next to the aluminium step wedge, and a digital radiograph was taken (Digora™ system; Soredex Orion Corporation, Helsinki, Finland). Radiographic images were obtained using the 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 (Digora™ Scanner) and analysed using Digo-ra™ for Windows 5.1 software.

### Dimensional change

Five Teflon® (Polytetrafluoroethylene, DuPont, HABIA, Knivsta, Sweden) moulds, 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 moulds

were filled with a slight excess of freshly mixed sealers and a microscope slide, also wrapped in cellophane, was pressed onto the upper surface of the mould. The assembled group was kept firmly joined with a C-shaped clamp and transferred to an incubator (37 °C, 95% RH) left to stand for a period corresponding to three times the setting time. After this period, the flat ends of the moulds, containing the samples, were ground with 600 grit wet sandpaper. The samples were removed from the mould, measured with a digital calliper, 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 on absorbent paper, and measured again for length. The percentage of the dimensional alterations was calculated using the formula:

$$\frac{L_{30} - L}{L} \times 100$$

where  $L_{30}$  is the length of the sample after 30 days of storage and  $L$  is the initial length of the sample.

### Solubility

A 1.5-mm-thick cylindrical Teflon® mould measuring 7.75 mm in inner diameter was filled with freshly

mixed sealer. The mould 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 mould and pressed manually in such a way that the plates touched the entire mould 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 setting time. As soon as the samples were removed from the mould, they were weighed three times each (HM-200, A & D Engineering, Inc., Bradford, MA, USA), and the mean reading 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). After this period, 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. Afterwards, they were weighed again. The weight loss of each sample (initial mass minus final mass), expressed as a percentage of the original mass ( $m\% = m_i - m_f$ ), was taken as the solubility of the sealer.

A volume of 7.5 mL of distilled water from each sample was poured into a cleaned and dried porcelain crucible. Each crucible was put into a muffle and burned at 550 °C. Ash was dissolved in 10 mL of a concentrated nitric acid (Merck KGaA, Darmstadt, Germany) using a glass stick. Following this, the samples were put into 50 mL volumetric flasks and the volume made up with ultrapure deionized water (MilliQ, Millipore, Billerica, MA, USA). The attained solutions were sprayed into the atomic absorption spectrophotometer (Perkin Elmer, Überlingen, Germany) to verify the presence of  $\text{Ca}^{2+}$ ,  $\text{K}^+$ ,  $\text{Zn}^{2+}$  and  $\text{Ag}^+$  ions. The arithmetic mean of three replicates for each specimen was recorded and considered as the result, expressed as  $\mu\text{g mL}^{-1}$ .

### Scanning electron microscopy examination

For scanning electron microscopy (SEM) examination, cylindrical Teflon® moulds (3 × 4 mm) were filled with freshly mixed sealers. The moulds were supported by a glass plate covered with a cellophane sheet and placed in a chamber (37 °C, 95% RH) for a period corresponding to three times the setting time. After that, the

samples were sectioned with a number 15 disposable surgical scalpel blade, fixed on a metallic stub (10 × 5 mm), and sputter-coated with gold–palladium (Bal-Tec AG, Balzers, Germany) at 20 mA. The morphologies of the external surface and the cross-section of the samples were qualitatively analysed under a field emission SEM (Jeol JSM 5410; Jeol Technic Co., Tokyo, Japan) at an accelerating voltage of 15 kV, a working distance from 6 to 10 mm, and at different magnifications.

### Statistical analysis

Five specimens from each group were tested and the means were statistically compared. The Kolmogorov–Smirnov 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% (SPSS 17.0 for Windows; SPSS Inc., Chicago, IL, USA).

## Results

### Setting time

The ANSI/ADA specification (2000) requires that the setting time of a sealer shall be within 10% of that stated by the manufacturers. According to them, the setting times of AH Plus, RSA RoekoSeal, and GuttaFlow are 480, 50, and 30 min, respectively. Conversely, the Activ GP manufacturer does not provide this information and only states that it has been formulated with an extended 15-min working time.

Statistical significant difference was observed between AH Plus ( $580.6 \pm 3.05$  min), GuttaFlow ( $24.0 \pm 2.0$  min), RoekoSeal ( $40.0 \pm 1.58$  min), and Activ GP ( $15.2 \pm 1.30$  min) groups ( $P < 0.05$ ). However, mean values showed agreement with the ANSI/ADA standardization (Table 2).

### Radiopacity test

AH Plus ( $6.0 \pm 0.12$  mmAl), GuttaFlow ( $3.0 \pm 0.04$  mmAl), and RoekoSeal ( $4.17 \pm 0.45$  mmAl) had radiopacity above the 3-mm of aluminium as recommended by ANSI/ADA Specification 57, while Activ GP ( $1.31 \pm 0.35$  mmAl) did not meet this requirement. Statistical analysis demonstrated difference between the experimental groups ( $P < 0.05$ ) (Table 2).

|                        | Experimental groups            |                           |                           |                                |
|------------------------|--------------------------------|---------------------------|---------------------------|--------------------------------|
|                        | I                              | II                        | III                       | IV                             |
|                        | AH Plus                        | GuttaFlow                 | RoekoSeal                 | Activ GP                       |
| Setting time (min)     | 580.6 ± 3.05 <sup>a</sup>      | 24.0 ± 2.0 <sup>b</sup>   | 40.0 ± 1.58 <sup>c</sup>  | 15.2 ± 1.30 <sup>d</sup>       |
| Radiopacity (mmAl)     | 6.0 ± 0.12 <sup>a</sup>        | 3.0 ± 0.04 <sup>b</sup>   | 4.17 ± 0.45 <sup>c</sup>  | <b>1.31 ± 0.35<sup>d</sup></b> |
| Dimensional change (%) | <b>1.34 ± 0.23<sup>a</sup></b> | 0.44 ± 0.16 <sup>b</sup>  | -1.33 ± 0.12 <sup>c</sup> | <b>1.95 ± 0.20<sup>d</sup></b> |
| Solubility (%)         | 0.36 ± 0.14 <sup>a</sup>       | -0.33 ± 0.14 <sup>b</sup> | 0.50 ± 0.10 <sup>a</sup>  | <b>11.8 ± 0.43<sup>c</sup></b> |

Values followed by different superscript lowercase letters in the horizontal differ among them by the *post hoc* Tukey–Kramer test ( $P < 0.05$ ). Values are mean ± standard deviation. Values in bold letters are in disagreement with ANSI/ADA requirements.

**Table 2** Physicochemical properties of endodontic sealers

### Dimensional change

ANSI/ADA standardization (2000) states that the mean linear shrinkage of the sealer shall not exceed 1% or 0.1% in expansion. Except for GuttaFlow (0.44 ± 0.16%), neither sealer conformed to the Specification 57 (ANSI/ADA 2000). All groups exhibited post-setting expansion except for RoekoSeal that showed shrinkage on setting (-1.33 ± 0.12%). Statistical analysis revealed significant difference between the experimental groups ( $P < 0.05$ ) (Table 2).

### Solubility

ANSI/ADA Specification 57 (2000) states that a root canal sealer should not exceed 3% by mass when the solubility of the set material is tested. Except for Activ GP (11.8 ± 0.43%) all sealers conformed to ANSI/ADA standardization ( $P < 0.05$ ) (Table 2). The deionized distilled water used for the solubility test and submitted to atomic absorption spectrometry showed a significant level of  $\text{Ca}^{2+}$ ,  $\text{K}^+$  and  $\text{Zn}^{2+}$  ions release in the Activ GP group compared with AH Plus, GuttaFlow, and RoekoSeal ( $P < 0.05$ ). Significant levels of  $\text{Ag}^+$  release were not observed in any group (Table 3).

**Table 3** Measurement of metal solubility of experimental groups by atomic absorption spectrometry, expressed as  $\mu\text{g mL}^{-1}$

| Ions             | Experimental Groups |             |             |             |
|------------------|---------------------|-------------|-------------|-------------|
|                  | I                   | II          | III         | IV          |
|                  | AH Plus             | GuttaFlow   | RoekoSeal   | Activ GP    |
| $\text{Ca}^{2+}$ | 1.81 ± 1.01         | 0.76 ± 0.12 | 1.31 ± 0.75 | 32.57 ± 5.0 |
| $\text{K}^+$     | 0.23 ± 0.04         | 0.33 ± 0.09 | 0.39 ± 0.15 | 1.57 ± 0.22 |
| $\text{Zn}^{2+}$ | <0.2                | 0.25 ± 0.05 | <0.2        | 8.20 ± 1.74 |
| $\text{Ag}^+$    | <1.0                | <1.0        | <1.0        | <1.0        |

### SEM examination

Selected photomicrographs of the polymerized samples obtained before and after water storage for 7 days are presented in Figs 1–4. Overall, it was noted that all surfaces had micromorphological changes after the solubility test. The loss of matrix was evident and the filler particles were more distinguishable. AH Plus (Fig. 1a,b), GuttaFlow (Fig. 2a,b), and RoekoSeal (Fig. 3a,b) groups had an external surface that appeared to be mostly homogeneous rough surfaces that decrease in roughness after the solubility test with a subsequent roughening of the surface. Cracking was not observed in the specimens except for the Activ GP group. The surface of Activ GP samples was relatively rougher when compared with the other groups and the surface material appeared to be more damaged after the solubility test (Fig. 4a,b).

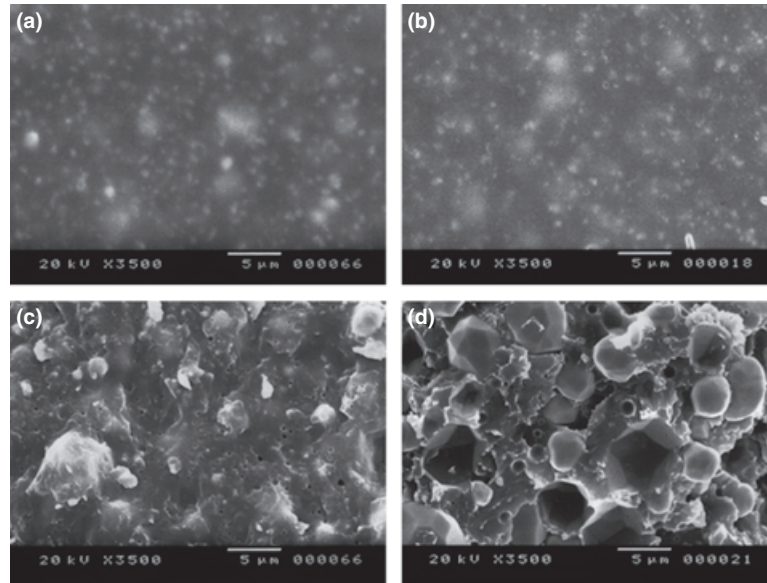
The scanning electron microscopy of the cross-section surface of the specimens revealed the presence of sphere-shaped polymers of different sizes that were nonhomogeneously dispersed. After the solubility test, the inner surface of AH Plus (Fig. 1c,d), GuttaFlow (Fig. 2c,d), and Activ GP (Fig. 4c,d) samples appeared to be much more irregular and rough. Several microcracks were seen at the filler-polymeric matrix interfaces in the Activ GP group before and after the solubility test (Fig. 4c,d). On the other hand, RoekoSeal group had a more uniform and compact layer comprised of sphere-shaped polymers with a higher amount of resin matrix (Fig. 3c,d).

### Discussion

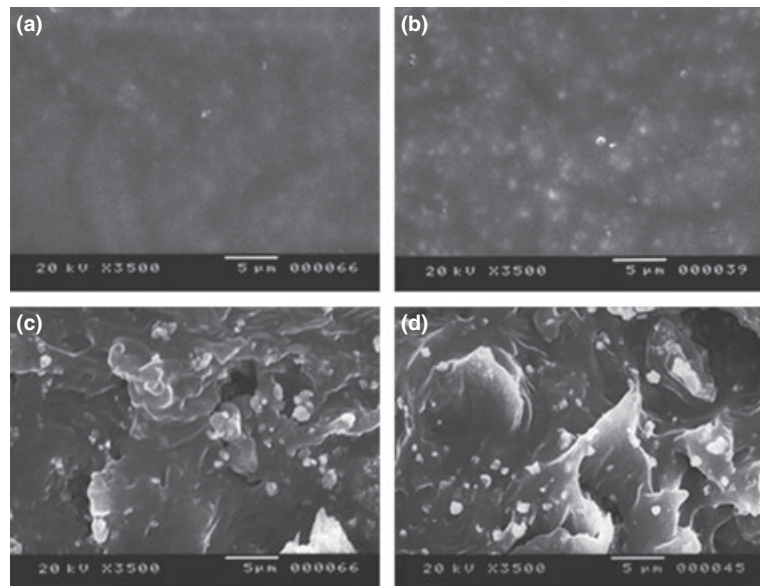
Setting time is primarily a control test on the stable behaviour of a product and is dependent on the constituent components, their particle size, the ambient temperature, and relative humidity (Ørstavik 1983, 2005, Ørstavik *et al.* 2001). In the present study, all



**Figure 1** Scanning electron micrographs of the morphological characteristics of AH Plus taken before and after water storage (3500 $\times$ , bar = 5  $\mu$ m). An external surface was observed that appeared to be mostly homogeneously rough (a) that decreased in roughness after the solubility test (b). Cracking was not observed. Before the test, the cross-section surface revealed the presence of sphere-shaped polymers nonhomogeneously dispersed (c) that appeared to be much more irregular and roughness after the test (d).



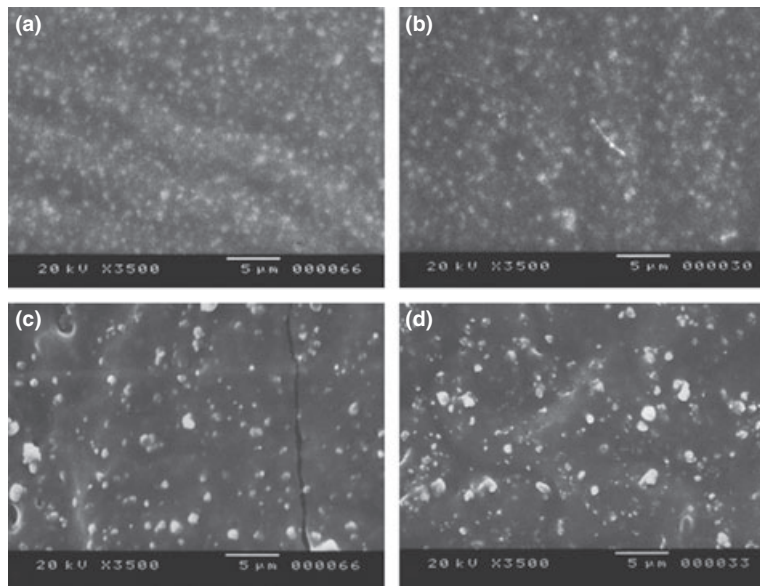
**Figure 2** Scanning electron micrographs of the morphological characteristics of GuttaFlow taken before and after water storage (3500 $\times$ , bar = 5  $\mu$ m). An external surface was observed that appeared to be mostly homogeneously smooth either before or after the solubility test (a and b, respectively). Cracking was not observed. Before the test, the cross-section surface revealed the presence of an irregular and nonhomogeneously dispersed layer (c) that increased in roughness after the test (d).



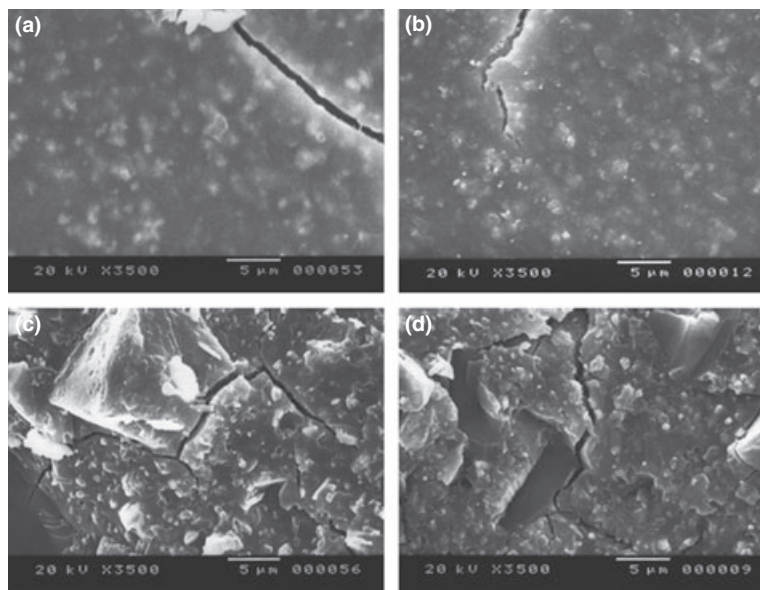
results were in accordance with ANSI/ADA specification. The longest setting time of AH Plus might be explained by this sealer being a two-component paste material based on a slow polymerization reaction of epoxy resin amines with high molecular weight (Bisphenol A and Bisphenol F), where the conversion of monomers into polymers occurs gradually (Lin-Gibson *et al.* 2006, Resende *et al.* 2009). These results were in agreement with previous research (Versiani *et al.* 2006, Resende *et al.* 2009), however, Garrido

*et al.* (2010) reported a setting time of 13 h to AH Plus, which is above the 10% variation permitted by the ADA's Specification Number 57.

The ANSI/ADA (2000) standards for the radiodensity of root canal sealing materials have been determined using conventional dental films where the radiographic images are obtained by the chemical processing (Taşdemir *et al.* 2008). However, digital radiography is becoming increasingly more common in the study of endodontic sealers (Baksi Akdeniz *et al.*



**Figure 3** Scanning electron micrographs of the morphological characteristics of RoekoSeal taken before and after water storage (3500 $\times$ , bar = 5  $\mu$ m). An external surface was observed that appeared to be mostly homogeneously smooth either before or after the solubility test (a and b, respectively). Cracking was not observed. The cross-section surface revealed the presence of a homogeneous, smooth and compact layer either before or after the solubility test (c and d, respectively).



**Figure 4** Scanning electron micrographs of the morphological characteristics of Active GP taken before and after water storage (3500 $\times$ , bar = 5  $\mu$ m). An external surface was observed that appeared to be mostly homogeneously rough (a) that decreased in roughness after the solubility test (b). Several microcracks were seen at the filler-polymeric matrix interfaces before and after the solubility test. Before the test, the cross-section surface revealed the presence of an irregular and nonhomogeneously dispersed layer (c) that increased in roughness after the test. Glass-ionomer fillers dislodged, leaving a rough, dimpled surface that was devoid of filler particles (d).

2007, Carvalho-Júnior *et al.* 2007a, Rasimick *et al.* 2007, Taşdemir *et al.* 2008). Thus, in the present study digital images of root filling materials and steps of an aluminium stepwedge were acquired using a phosphor plate system and a scanning, capturing and reading digital system.

An analysis of the composition of the tested materials revealed they all have radiopacifier agents (Table 1), except for Activ GP sealer. According to Tanomaru-Filho *et al.* (2008), the presence of barium aluminosil-

icate glass powder in its composition does not provide satisfactory radiopacity. AH Plus contains zirconium oxide, iron oxide and calcium tungstate as radiopacifiers (Tanomaru-Filho *et al.* 2007) and had the highest mean radiopacity value ( $6.0 \pm 0.12$  mmAl), as reported previously (Tagger & Katz 2003, Carvalho-Júnior *et al.* 2007a, Tanomaru-Filho *et al.* 2007, Taşdemir *et al.* 2008). RoekoSeal sealer contains zirconium dioxide as the radiopacifier (Baksi Akdeniz *et al.* 2007) and the present result ( $4.17 \pm 0.45$  mmAl) was

in agreement with previous findings that showed its radiopacity ranging from 4.0 to 5.7 (Tagger & Katz 2003, Gambarini *et al.* 2006, Tanomaru-Filho *et al.* 2007, Rasimick *et al.* 2007). The lower radiopacity of GuttaFlow ( $3.0 \pm 0.04$  mmAl) in comparison to RoekoSeal was also observed in previous studies (Tagger & Katz 2003, Gambarini *et al.* 2006, Rasimick *et al.* 2007, Tanomaru-Filho *et al.* 2007). As the manufacturer does not provide the chemical type and concentration of nano-silver in GuttaFlow, it is apparent that these particles did not exert a radiopaque characteristic.

High solubility of root canal sealers is undesirable because dissolution may cause the release of materials that could irritate periapical tissues and may also permit gaps to form between root canals and filling materials that is likely to increase bacterial leakage over time (Donnelly *et al.* 2007). Despite ANSI/ADA recommendations, in the present study a modification previously proposed for both tests was used, which achieved similar results with a decrease in the material volume necessary for the production of the test samples (Carvalho-Júnior *et al.* 2007b). In the present study, AH Plus expanded in the dimensional change test. The slow gain in mass over time was mostly due to the interaction energy between water and the adsorption sites for the system (i.e. a hydroxyl group, an amino group, the polymer chain end, etc.), filling the gaps between the polymer chains (McDermott 1993). Possibly, this expansion has compensated the shrinkage suffered by the resin-based sealer after polymerization (Phillips & Skinner 1991, Carvalho-Júnior *et al.* 2007b, Resende *et al.* 2009, Garrido *et al.* 2010).

The polymerization of the AH Plus is achieved through the polyamines monomers in its composition (1-adamantane amine, *N,N'*-dibenzyl-5-oxa-nonandiamine-1,9, TCD-Diamine). When the diepoxide compounds and polyamines paste are mixed together, the amine groups react with the epoxide groups to form a covalent bond. The utilization of amine groups aliphatic cyclic promotes modifications in the curing rate, cross-link density and morphology of epoxy resins. Each NH group can react with an epoxide group, so that the resulting polymer is heavily cross-linked, and is thus rigid and strong (McDermott 1993, Case *et al.* 2005). It may explain the homogeneous and compact rough surface revealed in SEM analysis and the low solubility results of AH Plus in the present study. Schäfer & Zandbiglari (2003) have also measured the solubility of epoxy resins (AH Plus, AH 26), silicone-based (RoekoSeal), calcium hydroxide (Apexit, Sealapex), zinc-oxide-eugenol (Aptal-Harz) and glass

ionomer-based (Ketac Endo) sealers in water and artificial saliva. According to these authors, AH Plus had the least weight loss of all sealers tested, independent of the solubility medium used.

Previous findings have shown that polydimethylsiloxane-based sealers expand slightly during setting and exhibited minimal water sorption and solubility (Donnelly *et al.* 2007, Monticelli *et al.* 2007). In agreement with this statement, in the present study GuttaFlow and RoekoSeal sealers had low solubility with minimum metal ion release. Considering the dimensional change test, GuttaFlow was the only sealer that conformed to the Specification 57 (ANSI/ADA 2000), underwent an expansion of approximately 0.44%. The same was not observed with its predecessor, RoekoSeal that demonstrated shrinkage (1.33%). Conversely, this result was not in agreement with Ørstavik *et al.* (2001) who reported that RoekoSeal had a small expansion of some 0.2% within 4 weeks, and was stable thereafter.

The presence of gutta-percha and nano-silver particles on GuttaFlow sealer gives better stability to the polymer matrix due to the attraction force between atoms of silver and molecules of gutta-percha into the polymer cross-link (Malynych *et al.* 2001). These interactions may explain the low silver ion release of GuttaFlow observed in atomic absorption spectroscopy. Besides, the presence of gutta-percha in the polymer cross-link of GuttaFlow resulted in higher water adsorption than RoekoSeal (Gong *et al.* 2008) explaining the differences on the results of the dimensional change test and the more uniform and compact layer observed on RoekoSeal surface under SEM.

The highest results of solubility and dimensional change of Activ GP sealer may be related to ANSI/ADA's methodology that recommends that a sample be immersed in water after a period of three times the setting time of the material. According to Wilson & McLean (1988), the setting time of the ionomer cement is completed only 24 h after mixing. During the maturation time of glass-ionomer cements, the presence of humidity may cause dissolution of anions and cations that form the matrix for areas surrounding the cement (Monticelli *et al.* 2007). This ion dissolution does not allow water, which is part of cement composition, to hydrate the matrix ( $\text{Ca}^{2+}$  and  $\text{Al}^{3+}$  form polysalts with groups of  $\text{COO}^-$  of polyacrylic acid), rendering it unstable (Carvalho-Júnior *et al.* 2003, Schäfer & Zandbiglari 2003, Donnelly *et al.* 2007). It could explain the rougher surface observed in SEM and the significant level of calcium, potassium and zinc ions



release of Activ GP in the present study. As glass ionomer cements' structures are likely to be influenced by the presence or absence of water, they are sensitive to dehydration early in the setting process (Wilson & McLean 1988). For SEM evaluation, dehydration of the sample is an essential process, which may explain the microcracks observed in the surface of Activ GP samples. Therefore, the results obtained with Activ GP in the present study should be interpreted with caution.

## Conclusions

Setting time of all sealers was in accordance with ANSI/ADA's requirements. Activ GP did not fulfill ANSI/ADA's protocols regarding radiopacity, dimensional alteration and solubility. GuttaFlow was the only sealer that conformed to the Specification 57 in all tests. SEM analysis revealed that all sealers' surfaces presented micromorphological changes after the solubility test.

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