

# Long-term sealing ability of Pulp Canal Sealer, AH-Plus, GuttaFlow and Epiphany

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## Abstract

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**Aim** To evaluate the long-term sealing ability of four contemporary endodontic sealers [Pulp Canal Sealer (PCS), AH-Plus, GuttaFlow and Epiphany] using a fluid filtration technique.

**Methodology** The palatal roots of 40 human maxillary molar teeth were selected and the root canal was prepared using a crown-down technique (apical size 40, 6% taper). Roots were irrigated with 3% NaOCl, 17% EDTA solution and rinsed with distilled water. Canals were filled with either PCS, AH-Plus, GuttaFlow or Epiphany using a single-cone technique ( $n = 8$ ). Twenty-four hours after filling, the

roots were connected to an automatic flow-recording device (Flodec<sup>®</sup> System) filled with double-distilled water under pressure (0.2 bar) to measure leakage. Flow rates were assessed at 6, 12 or 24-h and after 1-year of storage.

**Results** None of the materials fully prevented fluid flow. Fluid flow decreased after 6 h and decreased further after 12 h. After 24 h, PCS and AH-Plus allowed significantly more fluid flow than GuttaFlow and Epiphany. After 1 year, PCS allowed significantly more fluid flow than the other materials. No significant changes in leakage occurred between 24 h and 1 year.

**Conclusions** GuttaFlow and Epiphany allowed less fluid movement along filled straight roots.

**Keywords:** endodontic sealers, fluid filtration, microleakage, SEM.

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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 (Schilder 1967, Torabinejad & Pitt Ford 1996). However, the quality of the seal obtained with conventional zinc oxide–eugenol/gutta-percha materials is not perfect (Schafer & Zandbiglari 2003). Because these conventional materials do not effectively seal the root canal space, the outcome of root canal treatment is influenced by

the coronal seal that prevents re-infection (Saunders & Saunders 1994, Ray & Trope 1995). Using DNA bacterial signature techniques, Hommez *et al.* (2004) confirmed that in teeth with apical periodontitis, a poor coronal seal contributes to root canal re-infection. Yet, there is controversy about the full role of the coronal seal in successful therapy; many authorities consider that regardless of coronal seal, a complete seal of the root is required to maintain long-term periapical health (Tronstad *et al.* 2000). With this goal in mind, new endodontic sealers have been developed to improve the root canal seal beyond that currently possible with conventional materials.

AH-Plus (Dentsply Maillefer, Ballaigues, Switzerland) is an epoxy-based endodontic sealer that is used with gutta-percha in vertical or lateral compaction techniques. Although AH-Plus has adequate long-term dimensional stability, its sealing ability remains

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controversial partly because AH-Plus does not bond to gutta-percha (Ørstavik *et al.* 2001, Cobankara *et al.* 2002). Epiphany (Pentron Clinical Technologies LLC, Wallingford, CT, USA) is a dual-curing dimethacrylate resin that uses a primer. With this material, a thermo-plastic core material (Resilon, Pentron Clinical Technologies LLC, Wallingford, CT, USA) is bonded to the resin-based sealer, thereby establishing a so-called 'monoblock obturation' (Teixeira *et al.* 2004). Root canals filled with Epiphany exhibit less microleakage than roots filled with gutta-percha and conventional sealers (Shipper *et al.* 2004). GuttaFlow (Coltène Whaledent, Alstätten, Switzerland) consists of a mixture of gutta-percha powder, poly-dimethylsiloxane and silver particles. GuttaFlow flows readily at room temperature and expands slightly during setting. Clinically, GuttaFlow is used with gutta-percha points and compaction.

Dyes, glucose, bacteria and radioisotope tracers have all been used in laboratory models to assess microleakage after root sealing. Yet, some investigators have questioned the relevance of these tests to clinical conditions (Shemesh *et al.* 2006) because the size and shape of the tracers are different from bacteria and endotoxins that cause periapical disease and endodontic failure (Barthel *et al.* 1999). These tracer methods also have the limitation of being semi-quantitative. For these reasons, Wu *et al.* (1993, 1994) reported that the fluid filtration technique may be more appropriate to assess endodontic sealing strategies. Fluid filtration is based on the principle that no fluid movement will be detected if the root canal system is completely sealed. Further, the fluid filtration technique quantifies microleakage and allows repeated measurements because it is nondestructive (Camps & Pashley 2003). Up until now, fluid filtration tests of current endodontic sealers have been limited to short-term evaluations. The aim of the current study was to quantify long-term sealing properties of four contemporary root canal sealers using the fluid filtration technique.

## Materials and methods

### Canal preparation

Palatal roots from human maxillary first molars ( $n = 40$ ) were sectioned at the cervical level to obtain 10-mm long-sections of root. The canals in the roots were prepared and then root filled. The working length of each root was established with a size 15 K-file (Kerr, Biaggio, Switzerland) 0.5 mm short of the apical foramen. Roots were selected that had similar apical

foramen diameters prior to instrumentation. During root canal preparation, the canal space was enlarged using ProTaper instruments rotated at 250 rpm (Dentsply Maillefer) under a constant irrigation with 3% NaOCl. Apical patency was verified with a manual size 15 K-file between each ProTaper instrument. Preparation of the apical third was completed using nickel-titanium hand instruments (Dentsply Maillefer) to a size 40 with a 6% taper. After preparation was complete, the canal was irrigated with 17% EDTA for 1 min to dissolve the smear layer. The EDTA solution was neutralized with 3% NaOCl and then the canal was rinsed with distilled water (5 mL) and dried with paper points.

### Canal filling

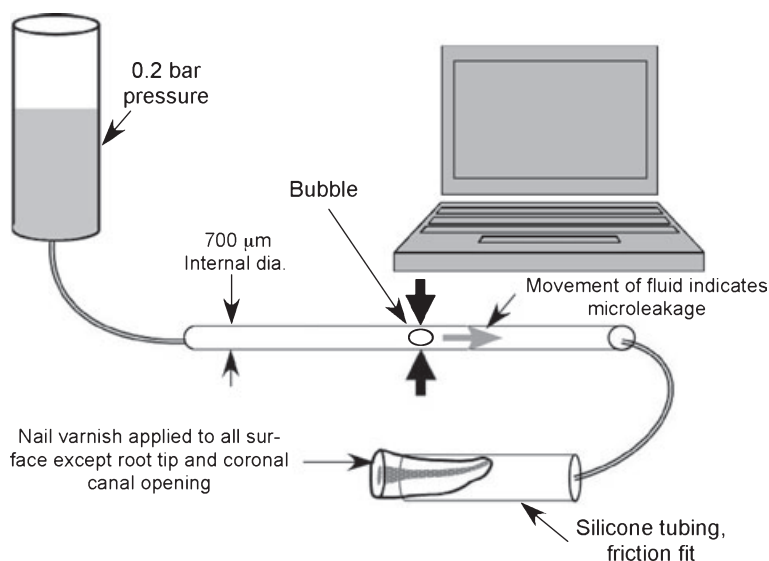
Endodontic sealers were mixed and used according to manufacturer's instructions and introduced into the canal space with a Lentulo spiral filler (Dentsply Maillefer, Ballaigues, Switzerland). For Pulp Canal Sealer (PCS), AH-Plus and GuttaFlow, calibrated gutta-percha points were placed at the working length and compacted vertically without heating the compacting instrument. For Epiphany, the self-etching primer was introduced into the canal with a 27-gauge needle (BD Microlance 3, Drogheda, Ireland) and left in place for 1 min before excess primer was removed with paper points. The Epiphany sealer was then introduced into the canal space and a Resilon point coated with sealer was inserted at the working length and compacted vertically. All specimens were then stored in a humid atmosphere for 24 h before testing for microleakage.

### Measurement of microleakage (fluid-flow technique)

Nail varnish was used to limit the passage of fluid across the dentinal tubules and assure that any fluid flow measured was caused by flow along the interface between the sealer and the dentine. The coronal end was not sealed by design to allow any leakage of the sealer to be detected at the end of the roots. The external surface of roots was double coated with nail varnish except at the tip of the root (1-mm apical surface free of varnish). Canals filled with gutta-percha (without sealer) and uncoated with varnish were used as positive controls ( $n = 8$ ); the same roots were then *entirely* covered with nail varnish for negative controls ( $n = 8$ ).

To measure fluid flow, the apical part of the root was cemented with cyanoacrylate (Zapit, Dental Ventures of America, Corona, CA, USA) to a thick-walled silicone tubing connected to an automated flow

**Figure 1** Experimental set-up used to measure fluid flow in endodontically treated roots. Each specimen was introduced into a soft silicone tubing and cemented with cyanoacrylate glue. The silicone tubing was connected to an automated flow recording device filled with double-distilled water under pressure ( $P = 0.2$  bar), after which fluid movement was detected with a Flodec® device. The detection limit of the Flodec® device was  $<5 \text{ nL min}^{-1}$  at 0.2 bar pressure.



recording device (Flodec® System; De Marco Engineering, Geneva, Switzerland; capillary diameter,  $700 \mu\text{m}$ ) filled with double-distilled water under a constant hydrostatic pressure (Fig. 1). Technical details about this measuring device have been reported previously (Ciucchi *et al.* 1995, 1997), but are summarized here. To minimize artefacts and allow long-term measurement of microleakage, thick-walled tubing was used to limit changes in volume from distortion of the tubing. The temperature of the apparatus was also strictly maintained at  $25 \text{ }^\circ\text{C}$  to avoid changes in volume from temperature. A  $+0.5$  bar pressure test was used to check for leaks prior to the beginning of the test and ultra-tight fittings were used throughout the system. The pre-applied pressure also forced the fluid into any voids present in the root-end fill. If leaks were observed in the fittings during this pre-test, measurements were aborted and leaks sealed. Such measures helped ensure that any subsequent fluid movement was due to leakage in the canal itself and not because of flaws in the technique.

To estimate microleakage, 0.2 bar water pressure was applied for 24 h to the roots and fluid flow was measured continuously. Microleakage in the negative controls was  $<0.01 \mu\text{L min}^{-1}$  (Table 1). The detection limit of the system was  $<5 \mu\text{L min}^{-1}$  (Ciucchi *et al.* 1997). At the end of the 24 h, specimens were stored in a humid atmosphere for 1 year at  $37 \text{ }^\circ\text{C}$ . During storage, dehydration and contamination of the specimens were limited by storing them in hermetically sealed jars containing 0.2% sodium azide. After 1 year, specimens were immersed in distilled water for 6 h before recording fluid-flow rates for 2 h.

### Scanning electron microscopy

To assess qualitatively what mechanisms might be responsible for leakage of the different sealers, scanning electron microscopy (SEM) was used. For each type of sealer, specimens that exhibited the greatest leakage were selected. These specimens were fractured longitudinally using a scalpel blade and hammer. They were

**Table 1** Fluid flow ( $\mu\text{L min}^{-1}$ ) measured at different time intervals for four endodontic sealing materials

Time	GP + varnish (negative control)	GP alone (positive control)	PCS	AH-Plus	Epiphany	GuttaFlow
0–6 h	$<0.01$	$0.24 \pm 0.07$ (8/8)	$0.20 \pm 0.13$ (7/8)	$0.17 \pm 0.05$ (4/8)	$0.10 \pm 0.05$ (5/8)	$0.08 \pm 0.03$ (3/8)
6–12 h	$<0.01$	$0.61 \pm 0.04$ (8/8)	$0.09 \pm 0.08$ (7/8)	$0.07 \pm 0.02$ (4/8)	$0.04 \pm 0.02$ (5/8)	$0.03 \pm 0.03$ (3/8)
12–24 h	$<0.01$	$0.19 \pm 0.01^a$ (8/8)	$0.04 \pm 0.02^b$ (7/8)	$0.04 \pm 0.06^b$ (4/8)	$0.01 \pm 0.02^b$ (5/8)	$0.02 \pm 0.02^b$ (3/8)
1 year	$<0.01$	n/a	$0.06 \pm 0.02^c$ (7/8)	$0.04 \pm 0.02^d$ (4/8)	$0.02 \pm 0.01^d$ (5/8)	$0.03 \pm 0.01^d$ (4/8)

Values are means  $\pm$  SD ( $n = 8$ ). Fraction of leaking specimens are indicated in parentheses (e.g. 7/8). For the 12–24 h and 1-year time intervals, values marked with different lower-case letters in the horizontal rows are significantly different (ANOVA, Tukey *post hoc* test,  $\alpha = 0.05$ ). The detection limit of the method was experimentally determined to be  $5 \mu\text{L min}^{-1}$ , rounded here to  $0.01 \mu\text{L min}^{-1}$ . GP, gutta-percha; PCS, Pulp Canal Sealer.

then fixed in 10% buffered formaldehyde overnight, processed through ascending alcohol concentrations to 100% alcohol and finally critical point dried. They were sputter coated with gold and examined in a Phillips XL 20 (Philips, Eindhoven, the Netherlands) scanning electron microscope.

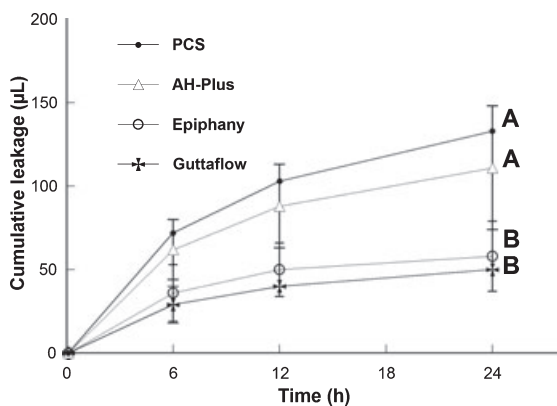
### Statistical analysis

One-way analysis of variance (ANOVA, Tukey *post hoc* analysis,  $\alpha = 0.05$ ) was used to compare mean cumulative leakages at 24 h and mean flow rates after 1 year. Two-sided paired *t*-tests ( $\alpha = 0.05$ ) were used to compare microleakage at 24 h and 1 year.

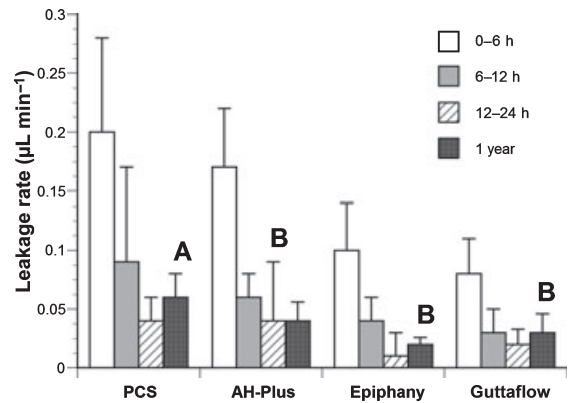
### Results

For negative controls (entirely coated with varnish), measurable fluid flow was not observed within the detection limits ( $0.01 \mu\text{L min}^{-1}$ ) of the model during the 24-h measurements (Table 1). As expected, specimens filled with gutta-percha only (positive controls) leaked significantly under pressure (Table 1). All materials allowed fluid to flow along the root dentine–sealer interface at all time intervals (Fig. 2). Fluid flow decreased after 6 h and decreased further after 12 h. After 24 h, PCS and AH-Plus exhibited significantly more cumulative leakage than GuttaFlow and Epiphany (ANOVA, Tukey *post hoc* test,  $\alpha = 0.05$ ).

When data were converted into flow rates ( $\mu\text{L min}^{-1}$ ), leakage rates were initially higher (0–6 h) than at later intervals (12–24 h). For PCS and AH-Plus,



**Figure 2** Cumulative leakage over 24 h measured by fluid filtration under pressure (0.2 bar). Fluid flow progressively decreased over time for all materials; Epiphany and GuttaFlow leaked significantly less than Pulp Canal Sealer (PCS) and AH-Plus. Error bars indicate SD ( $n = 8$ ). Letters indicate statistical groupings (ANOVA, Tukey,  $\alpha = 0.05$ ).



**Figure 3** Leakage rate ( $\mu\text{L min}^{-1}$ ) at four intervals for Pulp Canal Sealer (PCS), AH-Plus, Epiphany and GuttaFlow. Error bars indicate standard deviations ( $n = 8$ ). Letters indicate statistical differences between materials at 1 year (ANOVA, Tukey,  $\alpha = 0.05$ ). There were no statistical differences between the 24-h and 1-year flow rates for any material (two-sided, paired *t*-test,  $\alpha = 0.05$ ).

leakage rates were reduced by 80% after 24 h (Fig. 3). For GuttaFlow and Epiphany, leakage rates were decreased by 75% and 98%, respectively. After 24 h, there were no statistical differences between leakage rates (ANOVA, Tukey *post hoc* test,  $\alpha = 0.05$ ) and none of the materials produced a perfect seal (Fig. 2, Table 1).

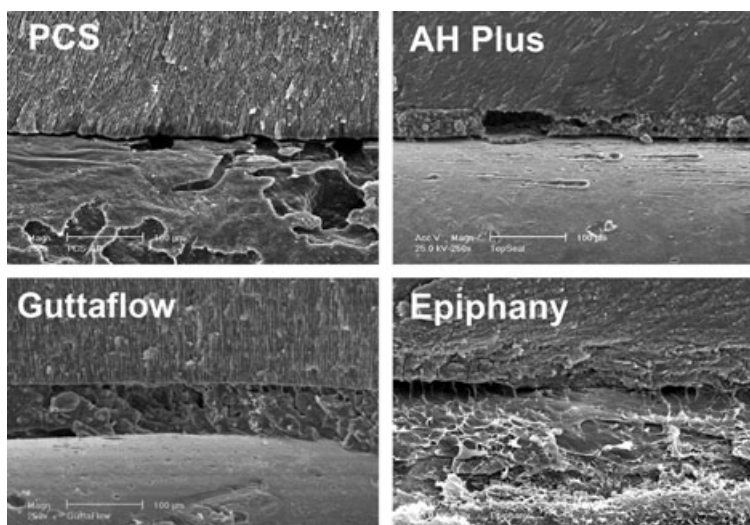
Pulp Canal Sealer exhibited significantly more leakage than the other materials after 1 year (ANOVA, Tukey *post hoc* test,  $\alpha = 0.05$ ). Although a trend towards increased leakage was observed (Fig. 3), there were no significant changes in leakage between 24 h and 1 year (two-sided paired *t*-test,  $\alpha = 0.05$ ).

Scanning electron microscopy was used to observe specimens that had the highest leakage after 24 h (Fig. 4). For PCS, the sealer contained continuous fluid-filled channels. For AH-Plus, there were gap-free regions between the dentine and the sealer but little adaptation between the sealer and the gutta-percha point. Porosities inside the sealer were also observed frequently. GuttaFlow had good adaptation to both root canal dentine and gutta-percha, but also had porosities inside the silicone-based material. Epiphany revealed a heterogeneous distribution of the resin-based sealer and a porous resin film, perhaps from an incomplete evaporation of the solvent.

### Discussion

The current study focused on the ability of materials to seal the apex because relative to the coronal root,

**Figure 4** Scanning electron micrographs (250×) of sealer–dentine interfaces for specimens that leaked the most. Voids were observed primarily in the Pulp Canal Sealer (PCS). Note that for PCS, the space between the sealer and dentine is a vacuum-induced artefact. In the AH-Plus specimen, debonding was observed between the sealer and gutta-percha cone. The GuttaFlow specimen had few voids. Hydrogel formation caused by incomplete solvent evaporation was observed in the Epiphany specimen.



apical sealing remains relatively vulnerable to microbial leakage (Galvan *et al.* 2002). Furthermore, apical leakage jeopardizes the biological response to endodontic sealers because it facilitates the release of unreacted components from materials directly into the periapical tissues (Bouillaguet *et al.* 2004).

The fluid filtration technique, originally described by Derkson *et al.* (1986), has been extensively used to measure dentine permeability, comparing the sealing properties of restorative materials, and evaluate the sealing ability of endodontic sealers. More recently, Wu *et al.* (2003) have used this technique to quantify coronal leakage after root filling. Other investigators have employed fluid filtration to measure the sealing ability of retrograde filling materials (Cobankara *et al.* 2002, Lamb *et al.* 2003).

In the current study, fluid flow was measured for 24 h to assess the performance of four contemporary endodontic sealers. The coronal end of the specimens was left open, and therefore fluid permeation was possible. This was done by design to simulate a failed coronal leakage situation that would assess the sealing ability of the root end. Previous studies (Yatsushiro *et al.* 1998, Sullivan *et al.* 1999) showed that negative controls may leak after relatively short (<10 min) periods. For this reason, early studies limited the duration of the measurement of fluid movement. However, more recent studies have measured leakage in root-end fillings over similar periods of time (e.g. 12–24 h) without evidence of instability (De Bruyne *et al.* 2005). In negative controls, the recordings reached equilibrium after approximately 5 min and were stable for 24 h. Several factors may have

accounted for the improved stability that was observed in the current study. For example, the pipette used with the automatic fluid flow recording device must be perfectly straight and centred inside the optical detector to allow the detector to follow the air bubble over the entire length of the pipette. To identify leaky or loose tubing connections, the pressure was initially increased to +0.5 bar to check the system before the main test was started. The dentine was sealed by using nail varnish and by placing the specimen inside the silicone tubing, to seal the external root surface (Fig. 1). Temperature changes, which also contribute to changes in volume of the water (and therefore to apparent fluid movement), were carefully controlled in the root, tubing and equipment throughout the test. The specimens were also placed into a closed environment (95–100% relative humidity) to avoid evaporative loss of water during measurements. Furthermore, the pressure applied (+0.2 bar) during the measurement of fluid flow in the current study was five times lower than the pressure used in the above-mentioned studies, which reduces the tendency of water to both evaporate and leak. In support of the results, negative controls used in other filtration models also have reported stable negative controls (Brackett *et al.* 2006, Stratton *et al.* 2006).

The results of the current study clearly demonstrate that none of the materials completely sealed the root apex *in vitro* (Table 1, Figs 2 and 3). Inadequate apical seals could result from the technique used to fill the canal system; for example, the use of a single-cone filling technique is often considered inferior to more sophisticated 3D compaction techniques. In the

single-cone technique, the volume of sealer is high relative to the volume of the cone, and this ratio promotes void formation and reduces the quality of the seal (Kontakiotis *et al.* 1997). However, it must be noted that the concept of the single-cone technique has been recently re-visited (Wu *et al.* 2006), and that the volume of the sealer used in the present study was minimized because gutta-percha and Resilon® cones were calibrated to the preparation. This was confirmed by SEM (Fig. 4). Use of the single-cone technique also allowed a comparison of the performance of all materials under relatively standardized conditions.

The observation that all sealers leaked throughout the 24-h test indicates that some voids are continuous and connect the apical and the coronal parts of the root. The decrease in leakage with time suggests that some voids are 'dead ends' and that flow decreases as these types of voids are progressively filled with water (Fig. 2).

Each material exhibited an architecture in SEM that seemed to correlate with its sealing performance (Figs 3 and 4). However, these observations should be interpreted cautiously, because gaps observed between the sealer and dentine may be fracture-induced artefacts. Amongst the materials tested, PCS and AH-Plus leaked the most. For PCS, high leakage could be explained by voids which are common with hand-mixed cements (Mutal & Gani 2005). Leakage of AH-Plus may have resulted from inadequate bonding between the sealer and the gutta-percha point, allowing fluid to flow at the interface. This possibility is in agreement with the results from Tay *et al.* (2005a). Recently, Sagsen *et al.* (2006) reported leakage rates for AH-Plus ( $0.0003 \mu\text{L min}^{-1} \text{cmH}_2\text{O}^{-1}$ ) comparable to those observed after 12 h in the current study ( $0.07 \mu\text{L min}^{-1}$  under 0.2 bar of pressure or  $0.0003 \mu\text{L min}^{-1} \text{cmH}_2\text{O}^{-1}$ ).

Leakage of GuttaFlow and Epiphany were less than AH-Plus and PCS. The low leakage observed for GuttaFlow was consistent with previous work (Bouillaguet *et al.* 2004). Furthermore, leakage rates reported for GuttaFlow at 24 h completely agree with those recently reported by Brackett *et al.* (2006). The results from the present study show  $0.02\text{--}0.03 \mu\text{L min}^{-1}$  under 0.2 bar of pressure, whereas Brackett *et al.* (2006) reported leakage rates of  $0.08 \mu\text{L min}^{-1}$  under 69 KPa of pressure. When converting pressure units and flow rates into hydraulic conductance values ( $\mu\text{L min}^{-1} \text{cmH}_2\text{O}^{-1}$ ) both studies show leakage values of  $0.015 \mu\text{L min}^{-1} \text{cm H}_2\text{O}^{-1}$  for GuttaFlow. This further supports the validity of the experimental set-up used in this study.

The relatively good performance of Resilon–Epiphany is congruent with other reports (Stratton *et al.* 2006, Tunga & Bodrumlu 2006). However, SEM indicates that bonding and sealing with Epiphany may not always be predictable (Fig. 4). Incomplete evaporation of the primer solvent in Epiphany promotes the formation of a hydrogel that is inherently leaky. On the other hand, hydrophilic primers, such as hydroxyethylmethacrylate, promote resin penetration in dentine tubules and more effective sealing (Nakabayashi & Takarada 1992). These complicating factors may contribute to a relatively high technique sensitivity experienced with Epiphany.

The sealing ability of all materials did not change significantly after 1 year of storage, but PCS leaked significantly more than any other material (Fig. 3). The reported expansion of AH-Plus over time may have facilitated its better long-term sealing ability (Ørstavik *et al.* 2001). The low leakage rate of GuttaFlow was in agreement with the results observed *in vitro* (Cobankara *et al.* 2002) and *in vivo* (Wu *et al.* 2006) with the initial version of this silicone-based material (RoeKoSeal). Epiphany performed well despite the possibility of enzymatic degradation (Tay *et al.* 2005b). However, the slight trend towards an increase in leakage observed for three materials after 1 year could indicate that degradation of the seal might occur. Alternatively, fluid under pressure might progressively increase the size of the internal voids or defects inside the root fill.

## Conclusions

The results support the use of the fluid filtration technique to evaluate the short- and long-term sealing ability of endodontic sealers. All materials exhibited some leakage, even after 1 year, but leakage rates decreased to 1-year levels after 24 h. Of the materials tested, GuttaFlow and Epiphany sealed the apex of straight roots most effectively. Further studies beyond 1 year *in vitro* or *in vivo* should help confirm the current results.

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