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Long-term sealing ability of Resilon apical root-end fillings

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Abstract

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Aim To evaluate *ex vivo* the long-term sealing ability of the SE Resilon Epiphany system as an apical root-end filling material.

Methodology A total of 60 standardized horizontal bovine root sections were divided into three groups filled with either gutta-percha with AH 26, tooth-coloured mineral trioxide aggregate (MTA) or Resilon pellets with Epiphany SE, and submitted to capillary flow porometry at 48 h, 1 and 6 months to assess the minimum, mean flow and maximum pore diameters. Results of the different materials and results by material and time were analysed statistically using nonparametric tests; the level of significance was set at 0.05.

Results Resilon had smaller pore diameters than gutta-percha and MTA at 48 h and smaller mean flow and maximum pore diameters than gutta-percha and MTA at 1 month. At 6 months Resilon had larger minimum pore diameters than gutta-percha. Although not always statistically significant, the minimum, mean flow and maximum pore diameters of gutta-percha and MTA diminished with time. This was not the case for Resilon, where the same parameters increased.

Conclusions All materials leaked at all times. Resilon performed better than gutta-percha and MTA in the short-term, but the seal of MTA and gutta-percha improved over time whereas the seal of Resilon deteriorated. It is critical to evaluate the performance of materials in the long-term contrary to most studies which are short-term.

Keywords: capillary flow porometry, Epiphany, leakage, Resilon, root-end filling, seal.

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Introduction

When orthograde root canal treatment is associated with post-treatment disease, surgical endodontics may be indicated. The procedure involves surgical debridement of pathological periradicular tissue, apical root-end resection, root-end cavity preparation and the placement of a root-end filling in an attempt to seal the root canal (Gutmann & Harrison 1994). The root-end filling should ideally produce a fluid-tight seal that prevents residual irritants and oral contaminants from exiting the root canal system and entering the periradicular tissues (Arens *et al.* 1998).

An ideal root-end filling material would adhere and adapt to the walls of the root-end preparation, prevent leakage of micro-organisms and their toxins into the periradicular tissues, be biocompatible, be insoluble in tissue fluids and dimensionally stable and remain unaffected by the presence of moisture (Arens *et al.* 1998). It is generally accepted that the most fluid-tight apical seal possible is required for successful periapical healing (Hirsch *et al.* 1979). If the seal is not fluid-tight, microleakage may occur. Leakage of various root-end filling materials has been investigated widely, mainly using dye penetration methods. However, there are certain disadvantages in using the linear measurement

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of dye penetration, including the destruction of the specimen, which makes further evaluation of samples impossible, and the lack of reproducible and comparable results (Schuurs *et al.* 1993, Wu & Wesselink 1993).

The reported pattern of leakage in endodontics differs according to the various techniques adopted (Wu *et al.* 2003). The fluid transport method was first reported by Greenhill & Pashley (1981) and adapted by Wu *et al.* (1993). This method investigates through-and-through voids and the result when using this technique indicates the diameter of the void. The dye penetration method investigates through-and-through as well as cul-de-sac voids and the result when using this technique indicates the length of the void rather than the diameter (Wu *et al.* 2003).

Capillary flow porometry which was first introduced in dentistry in 2005 (De Bruyne *et al.* 2005) is also used to evaluate through-and-through voids. This technique is used in membrane and filter media testing to measure through pores (Jena & Gupta 2002), as does the fluid transport method. In contrast to the fluid transport method, which gives an indication on the diameter of the void, CFP provides exact information on the diameter of the minimum, mean flow and maximum pore diameter at its most constricted part. The method has been approved by the American Society of Testing and Materials (1999) and was adapted successfully in collaboration with VITO (Flemish Institute for Technological Research, Mol, Belgium) to evaluate through pores in filled root canals or root sections (De Bruyne *et al.* 2005). The method also provides information on pore distribution.

A variety of substances have been proposed as root-end filling materials including amalgam, gutta-percha, zinc oxide–eugenol cements, dentine bonding agents, glass–ionomer cements, mineral trioxide aggregate (MTA) and other restorative materials (Gutmann & Harrison 1994). MTA shows excellent biocompatibility (De Bruyne & De Moor 2004) and, in spite of the limited clinical research, is considered by many clinicians as a standard during apical surgery (Nicholson *et al.* 1991, Asrari & Lobner 2003, Pistorius *et al.* 2003, Sousa *et al.* 2004). After the introduction of grey MTA a tooth-coloured or white MTA was introduced (Matt *et al.* 2004, Tselnik *et al.* 2004). Gutta-percha has been used frequently as a root-end filling material in the past and often the filling material is exposed apically when no root-end filling is placed. The Epiphany endodontic obturation system (Pentron, Wallingford, CT, USA) consists of Resilon obturation material available in points and pellets, and a dual-cure, hydrophilic resin sealer. The Resilon points or pellets can be processed in the same way as gutta-percha. Recently, a self-etch (SE) version of this sealer was introduced. Resilon material is a formulation of polymers of polyester with fillers and radiopacifers in a soft resin matrix. The pellets are used with a delivery system (Obtura-Spartan, Fenton, MO, USA). The manufacturer claims that after curing the combination of obturation material and sealer will create a monoblock in the canal that effectively resists leakage.

After periradicular surgery, the surface of the root-end filling is exposed to the periapical environment. Because of this exposure, decomposition of the material may occur and the seal of the filling may degrade. In order to obtain information on the performance of root-end filling materials on the long-term, the seal of root-end filling materials should be tested at different intervals after filling (Wu *et al.* 1998, De Bruyne *et al.* 2006).

The purpose of this study was to evaluate the sealing ability of the SE Resilon-Epiphany system as a root-end filling material and to compare it with warm gutta-percha and white MTA in standard bovine root sections at 48 h and after 1 and 6 months.

Materials and methods

Preparation and filling of root sections

Roots of freshly extracted bovine incisors with an external diameter of approximately 7 mm were selected and prepared into standardized sections 3 mm high. The central pulp lumen was drilled to 2.5 mm in diameter. For this purpose, the sections which were verified to have a natural internal diameter smaller than 2.5 mm were fixed in a clamp. A bur of 2.5 mm in diameter which was secured in a fixed position was passed once through the lumen.

Sixty of these sections were divided into three different groups and each group was filled according to the following scheme:

Group 1: warm gutta-percha (Obtura II, Obtura-Spartan) and AH 26 (Dentsply De Trey, Konstanz, Germany) (gutta-percha).

Group 2: Pro-Root MTA Tooth-Colored Formula (Dentsply Tulsa, Tulsa, OK, USA) (MTA).

Group 3: Resilon pellets (Pentron) (Obtura II delivery system; Obtura-Spartan) and Epiphany SE (Pentron) (Resilon).

The root sections were rinsed with physiological saline solution, dried with paper points and air spray and placed on a glass plate on top of a strip of polyester. All materials were mixed and handled according to the manufacturer's instructions and the root sections were filled. The filling materials were condensed with a plugger (RCPS 12P; Hu Friedy, Chicago, IL, USA) and excess material was removed. The root sections were kept for 24 h at a temperature of 37 °C and 95–100% relative humidity and then immersed in demineralized water for 24 h before measurement. After the first capillary flow measurement at 48 h the root sections were removed from the capillary flow porometer and stored in demineralized water at a temperature of 37 °C. They remained under these conditions except during the follow-up measurements that were undertaken at 1 and 6 months.

Measurement of capillary flow

Capillary flow porometry (CFP-1200-A; PMI, Ithaca, NY, USA) provides fully automated through pore analysis. A wetting liquid (Galwick: 15.9 Dynes cm^{-1}, PMI) was used to fill the pores of the sample. Because the wetting liquid's liquid/solid surface free energy is less than the solid/gas surface free energy, filling of the pores is spontaneous, but removal of the liquid from the pores is not. In order to remove the wetting liquid from pores and permit gas flow, pressure must be applied to the sample. The fully wetted sections were fixed in the sample chamber after which the sample chamber was sealed. Air was then allowed to flow into the chamber behind the sample. When the pressure reaches a point, it overcomes the capillary action of the fluid within the largest pore (maximum pore), and the sample's bubble point pressure is identified. After determination of the bubble point pressure, the pressure is increased and the flow is measured until all pores are empty, and the sample is considered dry. At this time the smallest or minimum pore has been identified. The mean flow pore is described as follows: half of the flow through a dry sample is through pores having a diameter greater than the mean flow pore diameter. The other half of the flow is through pores having a diameter smaller than the mean flow pore diameter. Pressure in CFP ranges from 0 to 200 psi or 1.4 MPa and the pore size range that can be measured lies between 0.035 and 500 μm. The flow meters detect the presence of pores by sensing the increase in flow rate due to emptying of pores. Differential pressures and flow rates through wet and dry samples are measured. Application of differential pressure on excess liquid on the sample causes liquid displacement. Measurement of the volume of displaced liquid allows computation of liquid permeability. The pore diameter (D) is derived from the following equation: $D = 4\ \gamma \cos \theta / p$ (γ = surface tension of the wetting liquid, θ = contact angle of the wetting liquid, p = differential pressure required to displace the wetting liquid from the pore) (Jena & Gupta 2003). All measurements were performed at VITO (Vlaamse Instelling voor Technologisch Onderzoek or Flemish Institute for Technological Research).

Statistical analysis

Results were analysed statistically using nonparametric tests. Comparisons were made between the leakage results of the different materials at 48 h, 1 and 6 months using Kruskal–Wallis tests; two by two analyses were performed by Mann–Whitney *U*-tests with Bonferroni correction.

Comparisons between the leakage results of each material at the specified time intervals were completed using Friedman tests and two by two comparisons were carried out by Wilcoxon Signed Ranks tests with Bonferroni correction. The level of significance was set at 0.05.

Results

Measurements were obtained for each sample at each point in time, confirming the presence of through pores regardless of which root-end filling material was being tested. Exact values for minimum, mean flow and maximum pore diameters of each sample were obtained.

The results of the study are summarized in Tables 1–3. For reasons of completeness the range and median of minimum, mean flow and maximum pore diameters of gutta-percha and MTA as reported in De Bruyne *et al.* (2006) are repeated in Tables 1–3.

Leakage results at 48 h, 1 and 6 months

From the Kruskal–Wallis tests and the Mann–Whitney *U*-tests with Bonferroni correction the following results were obtained. At 48 h significant differences between the minimum ($P < 0.001$), mean flow ($P < 0.001$) and maximum ($P < 0.001$) pore diameters could be demonstrated.

No significant differences between gutta-percha and MTA could be demonstrated but there were significant differences between gutta-percha and Resilon and between MTA and Resilon for minimum, mean flow and maximum pore diameters. At 48 h Resilon showed

Table 1 Range and median of minimum, mean flow and maximum pore diameters by root-end filling material at 48 h (μm)

		Minimum pore diameter (μm)		Mean flow pore diameter (μm)		Maximum pore diameter (μm)	
Group	Filling material	Range	Median	Range	Median	Range	Median
1	GP + AH 26	0.075–0.355	0.1995	0.141–0.395	0.2630	0.177–1.714	0.4375
2	MTA	0.070–0.258	0.2210	0.183–0.925	0.2760	0.193–1.304	0.4440
3	Resilon	0.082–0.201	0.1165	0.100–0.272	0.1465	0.127–0.433	0.2140

MTA, mineral trioxide aggregate.

Table 2 Range and median of minimum, mean flow and maximum pore diameters by root-end filling material at 1 month (μm)

		Minimum pore diameter (μm)		Mean flow pore diameter (μm)		Maximum pore diameter (μm)	
Group	Filling material	Range	Median	Range	Median	Range	Median
1	GP + AH 26	0.070–0.362	0.0875	0.106–0.455	0.2730	0.128–0.896	0.4410
2	MTA	0.070–0.330	0.2010	0.152–0.393	0.2880	0.162–0.854	0.4370
3	Resilon	0.069–0.198	0.1175	0.075–0.350	0.1525	0.088–0.432	0.2265

MTA, mineral trioxide aggregate.

Table 3 Range and median of minimum, mean flow and maximum pore diameters by root-end filling material at 6 months (μm)

		Minimum pore diameter (μm)		Mean flow pore diameter (μm)		Maximum pore diameter (μm)	
Group	Filling material	Range	Median	Range	Median	Range	Median
1	GP + AH 26	0.069–0.199	0.1060	0.077–0.302	0.1315	0.104–0.418	0.2200
2	MTA	0.069–0.216	0.1055	0.084–0.346	0.1490	0.111–0.818	0.2455
3	Resilon	0.083–0.240	0.1335	0.095–0.340	0.1685	0.106–0.402	0.2380

MTA, mineral trioxide aggregate.

smaller pore diameters than gutta-percha and MTA. The range and median of minimum, mean flow and maximum pore diameters at 48 h are shown in Table 1.

At 1 month there was no significant difference between the minimum pore diameters of the different materials, but significant differences between the mean flow ($P < 0.001$) and maximum ($P < 0.001$) pore diameters could be demonstrated. Concerning the mean flow and maximum pore diameters, no significant differences between gutta-percha and MTA could be demonstrated, but there were significant differences between gutta-percha and Resilon and between MTA and Resilon. At 1 month Resilon showed smaller mean flow and maximum pore diameters than gutta-percha and MTA. The range and median of minimum, mean flow and maximum pore diameters at 1 month are shown in Table 2.

At 6 months a significant difference between the minimum pore diameters could be demonstrated ($P < 0.05$), but there were no significant differences between the mean flow and maximum pore diameters of the different materials. Concerning the minimum pore diameters, there was a significant difference between gutta-percha and Resilon. No significant differences between gutta-percha and MTA and between MTA and Resilon could be demonstrated. At 6 months Resilon showed larger minimum pore diameters than gutta-percha. The range and median of minimum, mean flow and maximum pore diameters at 6 months are shown in Table 3.

Leakage results by material

From the Friedman tests the following results were obtained. Concerning the minimum pore diameters there were significant differences between the different points in time for gutta-percha and MTA, but not for Resilon. Results of the two by two comparisons are summarized in Table 4. Statistically significant

Root-end filling material		Friedman's test	Two by two comparisons		
			48 h vs. 1 month	48 h vs. 6 months	1 month vs. 6 months
GP + AH 26	Minimum pore diameter	*($P < 0.05$)		>	
	Mean flow pore diameter	*($P < 0.001$)		>	>
	Maximum pore diameter	*($P < 0.001$)		>	>
MTA	Minimum pore diameter	*($P < 0.01$)		>	>
	Mean flow pore diameter	*($P < 0.005$)		>	>
	Maximum pore diameter				
Resilon	Minimum pore diameter				
	Mean flow pore diameter				
	Maximum pore diameter				

MTA, mineral trioxide aggregate.

Table 4 Summary of significant differences (marked by an asterisk) between minimum, mean flow or maximum pore diameters at 48 h, 1 and 6 months and for two by two comparisons by material (> means the pore diameter is larger at the former than at the latter measurement)

decreases in size were found between 48 h and 6 months for gutta-percha and MTA and between 1 and 6 months for MTA.

Concerning the mean flow pore diameters there were significant differences between the different points in time for gutta-percha and MTA but not for Resilon. Results of the two by two comparisons are summarized in Table 4. Statistically significant decreases in size were found for gutta-percha and MTA between 48 h and 6 months and between 1 and 6 months.

Concerning the maximum pore diameters there were significant differences between the different points in time for gutta-percha but not for MTA and Resilon. Results of the two by two comparisons are summarized in Table 4. Statistically significant decreases in size were found for gutta-percha between 48 h and 6 months and between 1 and 6 months.

Discussion

Capillary flow porometry generates highly reproducible and accurate data (Gupta & Jena 1999). Therefore, because of its nondestructive nature and following a previous study (De Bruyne *et al.* 2006) CFP was chosen as the evaluation method for the present study. It provides, as the first and only method in leakage research, exact data on pore diameters which can be compared statistically and gives an indication whether bacteria or their metabolites will be able to pass through the sample. This is in contrast to other methods, which only compare materials without giving any information on the size of pores. As such, the method can overcome the problem of limited reproducibility and comparability of conventional methods for evaluating leakage (Wu & Wesselink 1993). CFP uses a wetting liquid with a low surface tension such that pores as small as 0.035 μm can be measured, which assures the detection of gaps of about 2 μm which were already observed between the root dentine and the Resilon primer. These gaps might be too small to be detected by, for example, bacterial penetration models (De-Deus *et al.* 2007).

The relatively high pressures used during CFP may be a concern. It needs to be emphasized, however, that during the present study and during all previous studies none of the fillings were dislodged. Results from a pilot study also showed that no statistically significant differences were evaluated between measurements when samples were measured multiple times immediately after each other (De Bruyne 2006). Apart from this the results from push-out tests revealed that micropush-out bond strengths of all materials tested were higher than the pressures used in the present study (Yan *et al.* 2006, Sly *et al.* 2007, Ureyen *et al.* 2008). This implies that the filling materials used in the present study will not be damaged during CFP.

As the purpose of the study was to compare root-end filling materials, standardized root sections were essential. Because human teeth are too small to be used to prepare standardized samples that are easy to handle, fix and evaluate in a reliable way, bovine teeth were used. As bovine teeth are easy to obtain and as the sections are large enough to adjust the central pulp lumen to the exact diameter, standardization is straightforward. Consequently cavities of equal size could be filled with different materials and compared under the same conditions, although these differ from the clinical situation. From the study of Nakamichi *et al.* (1983) it appeared that no statistically significant difference was found in adhesion of various materials to human or bovine dentine. Because of the larger diameter and same height, the C-factor in the present samples will be lower than in human teeth which results in less influence from contraction forces (Tay *et al.* 2005a).

As the manufacturer claims that after curing the combination of obturation material and sealer will create a monoblock that effectively resists leakage, it seemed that Resilon would be a perfect root-end filling material. It can be applied easily in the same way as gutta-percha, and the material sets fast, which is an advantage during surgery. Because of the similarity to gutta-percha and because of the fact that MTA is often considered as a standard of care, both these materials were selected as controls.

Similar to the results of previous studies performed with CFP on root-end fillings (De Bruyne *et al.* 2005), measurements were obtained for each sample at each time interval. The average length of bacteria varies between 0.2 and more than 10 μm, the width between 0.2 and 1.5 μm (Hobot 2002); and their metabolites are even smaller. Apart from this, one has to keep in mind the fact that bacteria are not rigid structures but can alter their outline. Therefore, in general the maximum pore diameter and the size of bacteria and their metabolites will be indicative of the possible leakage along the root-end filling materials. The minimum and mean flow pore diameters are relevant in terms of pore size distribution. Looking at the results, this means that some bacteria and definitely their metabolites will be able to pass along root end fillings.

At 48 h the minimum, mean flow and maximum pore diameters were smaller for Resilon than for gutta-percha and MTA. At 1 month this was not the case for the minimum pore diameter, but remained so for the mean flow and maximum pore diameters. At 6 months the difference for the mean flow pore and maximum pore diameters had disappeared, whereas at this time Resilon had larger minimum pore diameters than gutta-percha. Looking at the tables, although not always statistically significant, it appears that the minimum, mean flow and maximum pore diameters of gutta-percha and MTA diminished in the course of time, which was not the case for Resilon. For Resilon there was an increase. As the maximum pore diameter will determine the eventual seal of the material, this diameter is of major importance.

Until now improvement of the seal was seen over time for all materials tested by CFP (De Bruyne *et al.* 2006); Resilon seems to act differently. In a short-term study by Maltezos *et al.* (2006), which also tested Resilon as a root-end filling material, the bacterial leakage analysis (4-week observation) showed that Super-EBA leaked significantly more than Resilon and that there was no difference between Resilon and white Pro Root MTA. This is contrary to the present study where after 1 month Resilon still performed better than MTA. In contrast to the above, most other studies evaluated root fillings and not root-end fillings. In a recent study which evaluated short-term coronal leakage, Epiphany SE sealer and Resilon as a root filling was compared with gutta-percha with AH 26 or AH plus sealer using dye leakage and performed better (Bodrumlu & Tunga 2007a). Shipper *et al.* (2005) evaluated the prevention of apical periodontitis in an *in vivo* dog model and concluded that the Resilon 'Monoblock' System was associated with less apical periodontitis than gutta-percha with AH 26, maybe because of its superior resistance to coronal microleakage. In other studies on root fillings, Resilon performed better, equal or worse than gutta-percha (Shipper *et al.* 2004, Aptekar & Ginnan 2006, Biggs *et al.* 2006, Bodrumlu & Tunga 2006, 2007b, von Fraunhofer *et al.* 2006, Onay *et al.* 2006, Pitout *et al.* 2006, Sagsen *et al.* 2006, Shemesh *et al.* 2006, 2007, Stratton *et al.* 2006, Tunga & Bodrumlu 2006, Almeida *et al.* 2007, Baumgartner *et al.* 2007, De-Deus *et al.* 2007, Ishimura *et al.* 2007, Paque & Sirtes 2007, Raina *et al.* 2007, Silveira *et al.* 2007, Verissimo *et al.* 2007, Pasqualini *et al.* 2008), sometimes depending on the sealer used in combination with gutta-percha or the leakage assessment method. Interesting in the context of root-end fillings though is the fact that Resilon had significantly less leakage than gutta-percha with Grossman's cement in moist canals in a (short-term) study (Zmener *et al.* 2008). In surgical circumstances, which often are not ideal, this might be a major benefit. On the other hand, the biodegradation of Resilon (polycaprolactone) as mentioned by Tay *et al.* (2005b), but contradicted by Trope (2006) might also be of relevance.

Different from most studies which are short-term, Paque & Sirtes (2007) performed a long-term study using a fluid transportation model in which they showed that initially there was no difference in leakage between gutta-percha with AH Plus sealer and Resilon/Epiphany but after 16 months gutta-percha retained its seal whereas Resilon/Epiphany lost its sealing capacity. The results of the present study confirmed these long-term results. Different factors might contribute to this loss of seal over time. Although the configuration factor (C-factor) of the specimens in the present study was not as high as in root canals (Tay *et al.* 2005a) one or more bonded areas might pull off or debond in the course of time. De Munck *et al.* (2005), in their review on the durability of adhesion to tooth tissue, reported that after about 3 months all adhesives exhibited mechanical and morphological evidence of degradation, which probably will also be true for Epiphany SE. Colour discharge from the Resilon pellets, according to the manufacturer only food grade dye 'leaching out into the tooth', was seen in 11 samples at 1 month and in 13 samples at 6 months in the present study. This might also have contributed to the increased leakage (Shemesh *et al.* 2006). As it is not common during surgery, in the present study no EDTA was used to remove the smear layer as suggested by the manufacturer. Removing the smear layer might positively influence the results.

This evolution of the seal over time of Resilon is in contrast to the improvement of seal over time of gutta-percha and MTA, which has already been discussed extensively in a former study (De Bruyne *et al.* 2006). Changes in these root-end fillings occurred probably at the interface with the root dentine as the absence of voids within the materials was confirmed earlier (De Bruyne *et al.* 2005). Dimensional changes during time (Orstavik *et al.* 2001) and further hydration of MTA powder (Wu *et al.* 1998) are factors which might have contributed to the improvement of their seal.

Conclusion

Irrespective of the root-end filling material, each sample leaked along the filling material at all times. Resilon was unable to provide a fluid-tight seal. Whereas in the present study Resilon performed better than gutta-percha and MTA in the short-term, the seal of these materials improved over time whereas the seal of Resilon deteriorated. As not all materials evolve the same way, it is important to evaluate on the long-term basis contrary to most studies which are short-term.

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