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Long-term assessment of the seal provided by root-end filling materials in large cavities through capillary flow porometry

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Abstract

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Aim To evaluate the long-term sealing ability of a variety of materials when used as root-end fillings.

Methodology A total of 140 standardized horizontal bovine root sections (external diameter: 7 mm, height: 3 mm; internal diameter: 2.5 mm) were divided into seven groups, filled with either gutta-percha with AH26, Ketac Fil, Fuji IX, Tooth-Colored MTA, IRM, Ketac Fil with conditioner or Fuji IX with conditioner and submitted to capillary flow porometry at 1 and 6 months to assess minimum, mean flow and maximum pore diameters. Results of the different materials and results by material were analysed statistically using non-parametric tests; the level of significance was set at 0.05.

Results There were no significant differences between the minimum pore diameters associated with the materials at each time. At 1 month the mean flow pore diameters of Ketac Fil were significantly larger than those of gutta-percha, Ketac Fil with conditioner, Fuji IX with conditioner and IRM. There were significant differences between the maximum pore diameters at 1 month (all > IRM; Fuji IX > gutta-percha, Ketac Fil with conditioner, Fuji IX with conditioner) and 6 months (Fuji IX > gutta-percha, IRM; Ketac Fil > gutta-percha, IRM). There were significant differences in the minimum pore diameters between the different points in time for each material except IRM, in the mean flow pore diameters for each material and in the maximum pore diameters for each material except MTA.

Conclusions All materials were associated with capillary flow. IRM root-end fillings had through pores that were smaller than those associated with other materials. Conventionally setting glass–ionomer cements had the largest pores, although dentine conditioning improved their performance. The seal of all materials improved after 6 months.

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

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Introduction

Periradicular surgery 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

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unaffected by the presence of moisture (Arens *et al.* 1998). A wide range of materials have been proposed for this purpose, 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, 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 widely been investigated, mainly using dye penetration methods. However, there are certain disadvantages to using the linear measurement 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. The result when using this technique indicates the length of the void rather than the diameter (Wu *et al.* 2003).

Capillary flow porometry, a well-established method for evaluating 'through pores' in R&D and quality control outside dentistry, can also be applied in this field. This technique is often used in membrane and filter media testing, and can be used to measure through pores (Jena & Gupta 2002), as the fluid transport method does. 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 between tooth and filling materials (De Bruyne *et al.* 2005).The method provides exact information on pore sizes and pore distribution.

A variety of substances have been proposed for use as root-end filling materials. Both glass–ionomer cements and MTA show excellent biocompatibility (Nicholson *et al.* 1991, Asrari & Lobner 2003, Pistorius *et al.* 2003, De Bruyne & De Moor 2004, Sousa *et al.* 2004). Tooth-coloured or white MTA has only recently been introduced to the profession and, as a consequence, only limited research has been carried out on the properties of this material (Matt *et al.* 2004, Tselnik *et al.* 2004, De Bruyne *et al.* 2005). Glass–ionomers are being used as root-end filling materials with and without dentine conditioner, but removal of the smear layer is supposed to improve the seal of the material (Saunders & Saunders 1992, 1994). Within the group of conventionally-setting glass–ionomers, reinforced formulas are also available. Gutta-percha has been used frequently as a root-end filling material in the past and is often the filling material exposed apically when no root-end filling is placed. Reinforced zinc oxide-eugenol cements such as Super-EBA and IRM have been and are still being used regularly during periradicular surgery with good results (Niederman & Theodosopoulou 2003, Vasudev *et al.* 2003).

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. Therefore, the seal of root-end filling materials should be tested at different intervals after filling to reveal which material can provide a long-lasting seal (Wu *et al.* 1998).

The purpose of this study was to compare the sealing ability of MTA and two different glass–ionomer cements with gutta-percha with sealer and to reinforced zinc oxide-eugenol cement after 1 and 6 months. For the glass–ionomer cements, both normal and reinforced formulas were tested with and without the use of dentine conditioner. Specimens which had already been tested at 48 h using capillary flow porometry (De Bruyne *et al.* 2006) were retested using the same method.

Comparisons were made between the various materials at each time interval, as well as between the specified time intervals for each material.

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.

One hundred and forty of these sections were divided into seven different groups and each group was filled according to the following scheme:

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

Group 2: Ketac Fil Plus Aplicap (3M Espe, Seefeld, Germany) (glass–ionomer cement) (Ketac Fil).

Group 3: Fuji IX Capsules (GC-Corporation, Tokyo, Japan) (reinforced glass–ionomer cement) (Fuji IX).

Group 4: Pro Root MTA Tooth-Colored Formula (Dentsply Tulsa, Tulsa, OK, USA) (mineral trioxide aggregate) (MTA).

Group 5: IRM Caps (Dentsply Caulk, Milford, DE, USA) (reinforced zinc oxide-eugenol cement) (IRM).

Group 6: Ketac Fil Plus Aplicap (3M Espe) (glass–ionomer cement) with Ketac Conditioner (3M Espe) (Ketac Fil with conditioner).

Group 7: Fuji IX Capsules (GC-Corporation) (reinforced glass ionomer cement) with Cavity Conditioner (GC-Corporation) (Fuji IX with conditioner).

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. In groups 6 and 7, the smear layer was removed, using polyacrylic acid dentine conditioner. 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 12 P; 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 (De Bruyne *et al.* 2006), the root sections were removed from the capillary flow porometer and stored in demineralised 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, New York, USA) provides fully automated through pore analysis. A wetting liquid (Galwick: 15.9 dyn 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 (Fig. 1). 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 (Fig. 1). 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 (Fig. 1). At this time the smallest or minimum pore has been identified. The mean flow pore can be 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 capillary flow porometry ranges from 0 to 200 psi 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 increase in flow rate because of 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) can be derived from the following equation: $D = 4\gamma\cos\theta/p$, where γ is surface tension of the wetting liquid, θ, the contact angle of the wetting liquid and p, the differential pressure required to displace the wetting liquid from the pore (Jena & Gupta 2003). All measurements were performed at VITO.

Statistical analysis

Results were analysed statistically using non-parametric 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 Dunn tests.

Comparisons between the leakage results of each material at the specified time intervals were completed using Friedman tests and two by two comparisons were performed 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

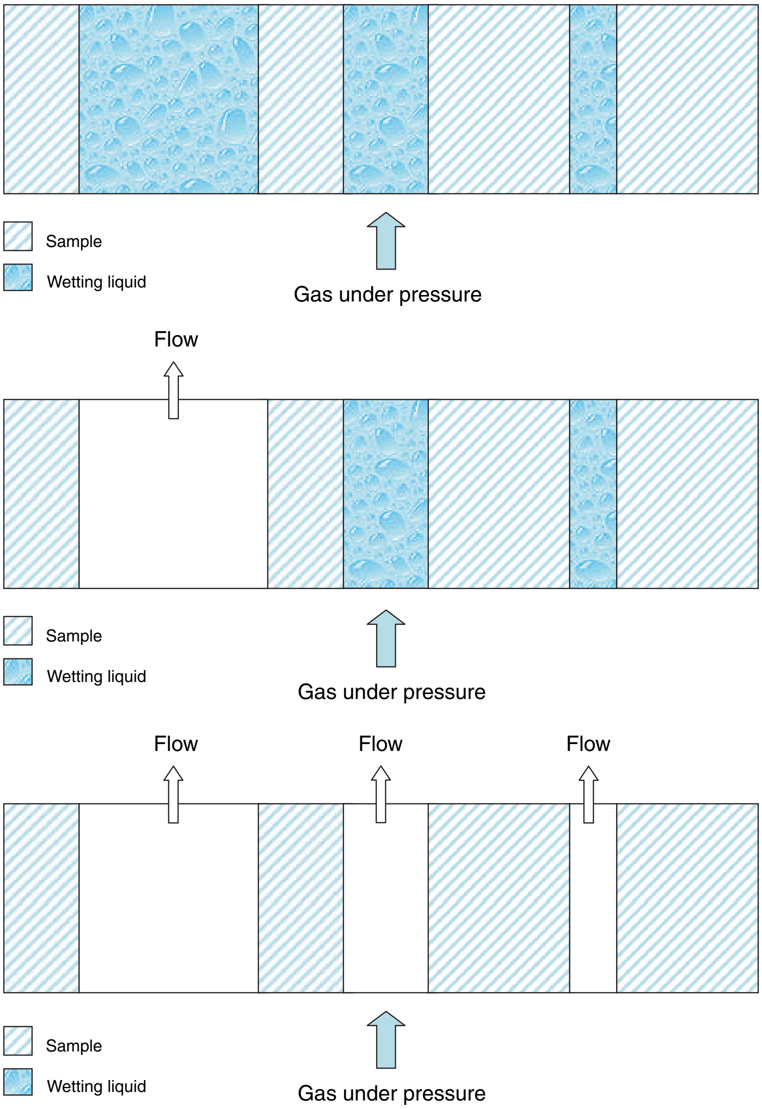

Figure 1 Principle of capillary flow porometry: as a result of gas pressure exerted on the sample (panel 1), the largest existing pore is emptied first through which now flow is measured (panel 2). Then in a descending order smaller pores will be emptied until all pores are empty (panel 3).

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 at 48 h as reported in De Bruyne *et al.* (2006) are summarized in Table 1.

Leakage results at 1 and 6 months

From the Kruskal–Wallis and Dunn tests the following results were obtained. At 1 month there was no significant difference between the minimum pore diameters of the different materials, but significant differences between the mean flow ($P < 0.05$) and maximum ($P < 0.001$) pore diameters could be demonstrated.

Concerning mean flow pore diameters, the medians of the leakage results can be arranged in the following descending order: Ketac Fil > MTA > Fuji IX > gutta-percha > Ketac Fil with conditioner > Fuji IX with conditioner > IRM. From the Dunn tests it appeared that there was a significant difference between Ketac Fil and gutta-percha, Ketac Fil with conditioner, Fuji IX with conditioner and IRM.

Concerning maximum pore diameters, the medians of the leakage results can be arranged in the following

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

		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	Ketac Fil	0.070–0.244	0.2040	0.139–0.613	0.2810	0.163–1.063	0.5160
3	Fuji IX	0.071–1.069	0.2100	0.272–1.396	0.6595	0.472–1.767	0.8610
4	MTA	0.070–0.258	0.2210	0.183–0.925	0.2760	0.193–1.304	0.4440
5	IRM	0.070–0.249	0.1480	0.132–0.390	0.2470	0.162–0.955	0.3650
6	Ketac Fil + C	0.085–0.300	0.2080	0.197–0.553	0.3125	0.306–0.697	0.4510
7	Fuji IX + C	0.093–0.351	0.2105	0.180–0.387	0.2740	0.313–0.798	0.4570

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

		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	Ketac Fil	0.070–0.342	0.2195	0.197–0.397	0.3505	0.308–1.090	0.4480
3	Fuji IX	0.072–0.313	0.1960	0.196–0.719	0.2760	0.258–1.285	0.5045
4	MTA	0.070–0.330	0.2010	0.152–0.393	0.2880	0.162–0.854	0.4370
5	IRM	0.070–0.315	0.1470	0.146–0.393	0.2125	0.161–0.436	0.3055
6	Ketac Fil + C	0.070–0.271	0.1970	0.159–0.378	0.2680	0.217–0.620	0.4395
7	Fuji IX + C	0.070–0.357	0.1755	0.121–0.392	0.2630	0.152–0.619	0.4360

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

		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	Ketac Fil	0.069–0.221	0.1190	0.074–0.357	0.1740	0.092–0.748	0.3320
3	Fuji IX	0.069–0.226	0.1420	0.078–0.923	0.1900	0.212–0.940	0.4320
4	MTA	0.069–0.216	0.1055	0.084–0.346	0.1490	0.111–0.818	0.2455
5	IRM	0.072–0.170	0.1075	0.098–0.211	0.1320	0.119–0.368	0.2105
6	Ketac Fil + C	0.069–0.209	0.1215	0.078–0.329	0.1660	0.114–0.559	0.2915
7	Fuji IX + C	0.068–0.216	0.1215	0.094–0.343	0.1555	0.126–0.459	0.2980

descending order: Fuji IX > Ketac Fil > gutta-percha > Ketac Fil with conditioner > MTA > Fuji IX with conditioner > IRM. From the Dunn tests it appeared that there was a significant difference between IRM and all other materials. Apart from this, there also was a significant difference between Fuji IX and gutta-percha, Ketac Fil with conditioner and Fuji IX with conditioner.

The range and median of minimum, mean flow and maximum pore diameters at 1 month are shown in Table 2.

At 6 months, one sample from group 2, three samples from group 3 and two samples from group 4 appeared to be broken and were discarded. There were no significant differences between the minimum and mean flow pore diameters of the different materials, but a significant difference between the maximum pore diameters could be demonstrated ($P < 0.001$).

Concerning maximum pore diameters, the medians of the leakage results can be arranged in the following descending order: Fuji IX > Ketac Fil > Fuji IX with conditioner > Ketac Fil with conditioner > MTA > gutta-percha > IRM. From the Dunn tests it appeared that there was a significant difference between Fuji IX and gutta-percha and IRM and between Ketac Fil and gutta-percha and IRM.

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 minimum pore diameters there were significant differences between the different points in time for each material except for IRM. Results of the two by two comparisons are summarized in Table 4. Statistically significant differences were found between 48 h and 6 months for gutta-percha, Ketac Fil, Fuji IX, MTA, Ketac Fil with conditioner and Fuji IX with conditioner, and between 1 and 6 months for Ketac Fil, Fuji IX, MTA, Ketac Fil with conditioner and Fuji IX with conditioner.

Concerning mean flow pore diameters there were significant differences between the different points in time for each material. Results of the two by two comparisons are summarized in Table 5. Statistically significant differences were found between 48 h and 1 month for Fuji IX, 48 h and 6 months for gutta-percha, Ketac Fil, Fuji IX, MTA, IRM, Ketac Fil with conditioner and Fuji IX with conditioner, and between 1 and 6 months for gutta-percha, Ketac Fil, MTA, IRM, Ketac Fil with conditioner and Fuji IX with conditioner.

Concerning maximum pore diameters there were significant differences between the different points in time for each material except for MTA. Results of the two by two comparisons are summarized in Table 6. Statistically significant differences were found between 48 h and 1 month for Fuji IX and Fuji IX with conditioner, 48 h and 6 months for gutta-percha, Ketac Fil, Fuji IX, IRM, Ketac Fil with conditioner and Fuji IX with conditioner, and between 1 and 6 months for gutta-percha, Ketac Fil and IRM.

Table 4 Summary of significant differences (marked by an asterisk) between minimum pore diameters at 48 h, 1 month and 6 months and for 2 by 2 comparisons by material

Root-end filling material	Friedman test	2 by 2 comparisons: 48 h–1 month	48 h–6 months	1–6 months
GP + AH 26	$P < 0.05$*		*	
Ketac Fil	$P < 0.001$*		*	*
Fuji IX	$P < 0.01$*		*	*
MTA	$P < 0.01$*		*	*
IRM				
Ketac Fil + C	$P < 0.005$*		*	*
Fuji IX + C	$P < 0.05$*		*	*

Table 5 Summary of significant differences (marked by an asterisk) between mean flow pore diameters at 48 h, 1 month and 6 months and for 2 by 2 comparisons by material

Root-end filling material	Friedman test	2 by 2 comparisons: 48 h–1 month	48 h–6 months	1–6 months
GP + AH 26	$P < 0.001$*		*	*
Ketac Fil	$P < 0.001$*		*	*
Fuji IX	$P < 0.005$*	*	*	
MTA	$P < 0.005$*		*	*
IRM	$P < 0.001$*		*	*
Ketac Fil + C	$P < 0.001$*		*	*
Fuji IX + C	$P < 0.01$*		*	*

Table 6 Summary of significant differences (marked by an asterisk) between maximum flow pore diameters at 48 h, 1 month and 6 months and for 2 by 2 comparisons by material

Root-end filling material	Friedman test	2 by 2 comparisons: 48 h–1 month	48 h–6 months	1–6 months
GP + AH 26	$P < 0.001$*		*	*
Ketac Fil	$P < 0.001$*		*	*
Fuji IX	$P < 0.001$*	*	*	
MTA				
IRM	$P < 0.001$*		*	*
Ketac Fil + C	$P < 0.005$*		*	
Fuji IX + C	$P < 0.001$*	*	*	

Discussion

Capillary flow porometry was chosen as the evaluation method because of its non-destructive nature and the highly reproducible and accurate data it generates (Gupta *et al.* 1999, Mayer 2002). As such, the method can overcome the problem of limited reproducibility and comparability of conventional methods for evaluating leakage (Wu & Wesselink 1993, Pommel & Camps 2001)

As the purpose of the study was to compare different root-end filling materials, standardized root sections were needed. Because human teeth are too small to be used to prepare standardized samples that are easy to handle, bovine teeth were used. Consequently cavities of equal size could be filled with different materials and compared under the same conditions, although these differ from the clinical situation. Root-end filling materials are also used when restoring perforation and root resorption defects. In such cases the affected area is often larger and comparable to the size of the cavities in the bovine samples. From the study of Nakamichi *et al.* (1983) it appeared that no statistically significant difference was found in adhesion of various materials, including a glass–ionomer cement, to human or bovine dentine, although the mean values were always slightly lower with bovine teeth. Thus the use of bovine dentine might have influenced the results.

Similar to the results of a previous study performed with capillary flow porometry 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. 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 size of the minimum pore diameters seemed to be of the same order for each material: there were no significant differences between the materials at 48 h, 1 and 6 months. On the other hand, there were significant differences between the materials for the mean flow pore diameter at 48 h and 1 month and for the maximum pore diameter at all times. The mean flow pore diameter seems to be of less importance in the present study. As the maximum pore diameter will determine the eventual seal of the material, these differences are of major importance. IRM at all times had the smallest maximum pore diameter, although another leakage study (fluid transport method) found that IRM leaked more than MTA (Fogel & Peikoff 2001). Both studies measured through pores, but the method differed. The study by Kazemi *et al.* (1993) mentioned continuous dimensional loss of zinc oxide-eugenol materials. In the present study this did not seem to influence the results of IRM Caps, which have a standardized powder to liquid ratio. When the material is hand-mixed, the ratio might have an influence on microleakage and possible dimensional losses (Crooks *et al.* 1994).

Glass–ionomer cements appeared to have larger maximum pores than the other materials, the reinforced glass–ionomer cement showing the largest ones. When glass–ionomers were used after dentine conditioning, the maximum pore diameters diminished. These results are in contrast to those of a previous study (De Bruyne *et al.* 2005) where Fuji IX without conditioner performed better than MTA and IRM. Similar to the previous study and contrary to the clinical situation, root-end fillings were placed under ideal circumstances, with excellent visibility and no moisture contamination. The size of the root-end cavity differed however, and it is unclear whether this might have influenced the results. Apart from this, the results suggest the use of dentine conditioner before glass–ionomer cements are placed as root-end filling materials. On the other hand, in a clinical situation, the low pH of dentine conditioners must be taken into account because of possible damage to the surrounding tissues (Bruce *et al.* 1993, Kinomoto *et al.* 2003).

Evaluation of the different materials showed that the seal improved over time. For either minimum, mean flow or maximum pore diameters, the seal of each material was better at 6 months. In most cases, the seal at 1 month was also better than the seal at 48 h, and the seal at 48 h was never significantly better than the seal at 1 month. This suggests that until 6 months after placement, loss of the seal as a result of decomposition of the root-end filling material, does not seem to be a major issue for any of the materials. This result is confirmed in other studies in which reduced leakage over time was also observed (King *et al.* 1990, Inoue *et al.* 1991, Wu *et al.* 1998, Greer *et al.* 2001). The results of the present and former studies imply that changes in the root-end filling occur probably at the interface with the root dentine, as a former study (De Bruyne *et al.* 2005) has confirmed the absence of voids within the material. Several explanations for these changes are possible. Dimensional changes of materials over time (Feilzer *et al.* 1995, Ørstavik *et al.* 2001) as well as storage conditions (Cattani-Lorente *et al.* 1994, Feilzer *et al.* 1995) and the complex ageing mechanisms of glass–ionomer cements (Cattani-Lorente *et al.* 1994) may have influenced the results and contributed to the improvement of the seal. Furthermore, it is assumed that, in moisture, further hydration of MTA powder may result in an increase in compressive strength and a reduction in leakage (Wu *et al.* 1998). Although the mechanisms behind them are not clear, the changes cause a reduction in leakage and consequently provide a better seal.

Based on the present data, IRM performed better than glass–ionomer cements. It is, however, important to realize that apart from sealing capacity, other factors may also influence the clinical performance of root-end fillings. This study was performed *in vitro* in the most ideal circumstances for root-end filling materials: no moisture contamination and optimal visibility. Biocompatibility is another very important factor and will influence the clinical performance of any root-end filling material.

Conclusion

Irrespective of the root-end filling material, each sample leaked along the filling material at 1 and 6 months. Thus none of the materials was able to provide a fluid-tight seal.

Through pores in IRM root-end fillings appeared to be smaller than in all other materials tested. Conventionally setting glass–ionomer cements had larger through pores than other materials, but dentine conditioning improved their performance. When evaluated in the long term, the seal of all materials improved after 6 months.

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