Presence of pores and vacuoles in set endodontic sealers

L. Mutal & O. Gani

Department of Endodontics, Faculty of Dentistry, National University of Córdoba, Córdoba, Argentina

Abstract

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Aim To assess qualitatively the presence of pores and vacuoles in the structure of various endodontic sealers when set.

Methodology Eight specimens were prepared in keeping with Instituto Argentino de Racionalización de Materiales (IRAM) and ISO regulations for each of the 10 sealers assessed. Four specimens per sealer were examined to identify the presence of structural defects, termed pores, on the external surface. The remaining four specimens were used to examine the presence of defects on the surface exposed by cross-sectional fracture; these were termed vacuoles. The largest and smallest diameters of the pores and vacuoles were measured by scanning electron microscope on both surfaces. The structural defects were classified according to their frequency as abundant, frequent, scarce or exceptional.

Results Pores and vacuoles were consistently found in every specimen of each sealer. However, their frequency and dimensions were greater in zinc-eugenol-based sealers than in epoxy-resins and glass–ionomer sealers; they increased if the sealer contained calcium hydroxide. The diameter of the pores ranged from 5 to 320 μm and the diameter of the vacuoles ranged from 80 to 500 μm. The diameter of the vacuoles always exceeded that of the pores.

Conclusions Pores and vacuoles were a consistent finding in set sealers. Their frequency and size depended on the density of the sealer and increased when the sealers contained calcium hydroxide.

Keywords: endodontic obturation, endodontic sealers, porosity, root canal obturation, vacuolar structure, vacuoles.

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Introduction

The role of an endodontic sealer in the lateral condensation technique is to cement the gutta-percha cones together and to the walls of the root canal in the hope of creating a compact, stable and impermeable filling (Ingle & West 1998).

Higginbotham (1967), Wennberg & Ørstavik (1990) and Wesselink (1990) amongst others have reported that the efficacy of the sealer increases as the cement film thickness reduces. However, Georgopoulou *et al.* (1995) observed that the sealing ability might depend on the type of cement employed; their studies showed that AH 26 and Sealapex created a better seal in thick layers (0.3 mm) whereas Ketac-Endo, Roth 801 and Tubli-Seal EWT created a better seal in thin layers (0.05 mm). Furthermore, studies by Wiener & Schilder (1971), Peters (1986) and Kazemi *et al.* (1993) provided evidence that dimensional changes and variations in solubility would significantly affect the performance of sealers, and recommended a reduction in cement film thickness for improved efficacy.

According to Wu *et al.* (1995), sealer permeability might change over time and seems to depend on several factors including the thickness of the sealer layer and solubility of the material. These authors also reported that the performance of sealers at thicknesses ranging from 0.05 to 0.25 mm might have clinical relevance.

Correspondence: Liliana Mutal, El Plumerillo 2438, Parque Chacabuco, 5008 Córdoba, Argentina (Tel.: +543514808443; fax: +543514235385; e-mail: lilimutal@hotmail.com).

McElroy (1955) reported that a porous material was not a safe sealer for root canals. At the same time, Grossman (1957) described the desirable physical properties of an obturation material but failed to refer explicitly to structural aspects. With very few modifications, the requirements he reported still hold true.

Several studies have addressed different aspects of the properties and characteristics of sealers such as their granularity (Caicedo & von Fraunhofer 1988, Valera *et al.* 2000), the dimensional changes they undergo after setting (Wiener & Schilder 1971, Kazemi *et al.* 1993), their solubility (Peters 1986, Wu *et al.* 1994, 1995, Kaplan *et al.* 1997) and sealing capacity (Higginbotham 1967, Wiener & Schilder 1971, Branstetter & von Fraunhofer 1982, Caicedo & von Fraunhofer 1988). However, few reports have addressed the structural features of this group of materials. Apical permeability has been extensively studied, but remarkably little attention has been given to the causes of such permeability.

Only limited information is available on the presence of pores and vacuoles within the sealer mass. This aspect is of importance because of the potential detrimental effect on the performance of sealers that are in permanent contact with periradicular fluids.

The aim of the present study was to perform a qualitative study on a variety of structural aspects of set endodontic cements using scanning electron microscope (SEM). Particular emphasis was placed on the evaluation of pores and vacuoles.

Materials and methods

To evaluate the structure of set endodontic cements, 10 endodontic sealers (Table 1) that differed in chemical composition and, in one case, in the mode of preparation prior to use, were included in the study. The study involved the assessment of surface defects such as orifices, concavities, craters, etc. that we grouped under the term 'pores' and defects that appeared on the fracture surface of the specimens which we referred to as 'vacuoles'.

The experiment was performed *in vitro* under standardized conditions in keeping with the regulations of the Argentine Institute of Rationalization of Materials (Instituto Argentino de Racionalización de Materiales – IRAM) 27048/94 and ISO 6876/86. Eighty plastic rings, 20 mm in diameter and 1.5 mm in height, coated with copper and chromium deposited by an electrochemical technique, were divided into groups of eight and placed on glass slabs. The rings were filled with the corresponding sealer under study.

Table 1 List of sealers included in the study

Zinc-eugenol-based sealers
Pulp Canal Sealer Cement (Kerr-Sybron, Romulus, MI, USA)
Tubli-Seal EWT (Kerr-Sybron)
Endomethasone (Septodont, St Maur, France)
Roth 801 Cement (Roth International Ltd, Chicago, IL, USA)
Glass–ionomer sealer
Ketac-Endo (ESPE-Gmbh Co., Seefeld, Berby, Germany)
Resin sealers
AH 26 (Dentsply, De Trey Konstanz, Germany)
AH Plus (Caulk; Dentsply, Milford, DE, USA)
Sealers containing calcium hydroxide
Apexit (Vivadent, Schaan, Liechtenstein, Germany)
Sealer 26 (Dentsply, Petrópolis, Brazil)
Sealapex (Kerr-Sybron)

A single operator prepared the samples in keeping with the instructions of the manufacturers in terms of proportions, times, mixing techniques, etc. Once the mixed sealer was ready, a spatula was used to fill a ring with the sealer. The spatula was placed vertically in the centre of the ring allowing its tip to touch the glass slabs. By gravity the sealer flowed down and filled the ring. No vibration or pressure was applied.

The sealers were allowed to set at 37 °C and at least 95% relative humidity. Sealer hardening was confirmed by visual inspection. In keeping with Allan *et al.* (2001), a 27-gauge needle was inserted every 30 min during the first 8 h and less frequently thereafter to evaluate surface set at varying times. The sealer was considered to have set when the needle did not adhere or leave an imprint.

The evaluation of setting was carried out at the periphery of the core material that contacted the ring to avoid potential alteration in the external and/or internal structure(s) of the samples induced by the contact, pressure or force of the indenters.

Subsequently, each group made up of eight samples and identified by the name of the sealer was subdivided into subgroups A and B having four samples each. Subgroup A was used to study the structure of the external surface of the specimens and subgroup B was employed to study the structure of the fractured surfaces. As the specimens were fragile, the fracture was performed manually.

The study of both surfaces was performed by SEM. The samples were coated with gold–palladium (Au–Pd), 400–500 Å for a working tension of 5–10 kV.

A team of two operators analysed the samples. They performed an initial overall assessment at a magnification of ×160 to establish a gross estimate of the frequency of pores and vacuoles. The quantitative evaluation was based on the following categories and the consistency of the findings for the four samples corresponding to each group: abundant (XXXX), when at a magnification of ×160 the structural defects were found in all the observed fields; frequent (XXX), when at a magnification of ×160 the structural defects failed to appear in some of the observed fields; scarce (XX), when meticulous observation of each of the surfaces was required to detect the structural defects; exceptional (X), when structural defects were a rare finding.

At a magnification of ×320 each surface was analysed in more detail. Photographs were taken and employed to measure the diameter of pores and vacuoles. The most representative areas in the central, more reliable area of each block were selected.

Results

The cements Roth 801 and Sealapex were discarded because they exhibited setting flaws; the remaining cements set adequately. The surfaces were either smooth or granular and featured orifices or concavities of varying shapes and sizes. As described previously, when these defects were found on the external surface they were termed as pores and when they were found on the fracture surface of the specimen they were termed as vacuoles. The most relevant data on the dimensions, frequency and other structural characteristics that resulted from the analysis of the 'external surface' (ES) and the 'fracture surface' (FS) of each of the sealers are described below and presented in Table 2.

1. *Pulp canal sealer cement*: ES: abundant granules on the surface. Abundant pores approximately 5 μm in diameter. FS: smooth. The abundant vacuoles were up to 120 μm in diameter (Fig. 1a,b).

2. *Tubli-seal EWT*: ES: smooth, cracked surface with some granules scattered on the surface. Scarce pores approximately 5 μm in diameter. FS: smooth with scarce granules. Abundant vacuoles of varying sizes, up to 260 μm in diameter. The surface resembled the cracked external surface (Fig. 2a,b).

3. *Endomethasone*: ES: the surface was fully covered by large granules. Scarce funnel-like pores in the surface were up to 90 μm in diameter. FS: compact appearance. Vacuoles up to 200 μm in diameter were a frequent finding.

Table 2 Maximum dimensions and frequency of pores and vacuoles detected in the structure of different sealers after setting

Sealer	Pores (diameter; μm)	Incidence	Vacuoles (diameter; μm)	Incidence
Pulp Canal Sealer Cement	≈5	XXXX	120	XXXX
Tubli-Seal EWT	≈5	XX	260	XXXX
Endomethasone	90	XX	200	XXX
AH 26	≈5	X	80	XXX
AH Plus	≈5	X	80	XX
Ketac-Endo	≈10	X	100	XXXX
Apexit	80	XXXX	120	XXXX
Sealer 26	320	XXXX	500	XXXX

XXXX, abundant; XXX, frequent; XX, scarce; X, exceptional.

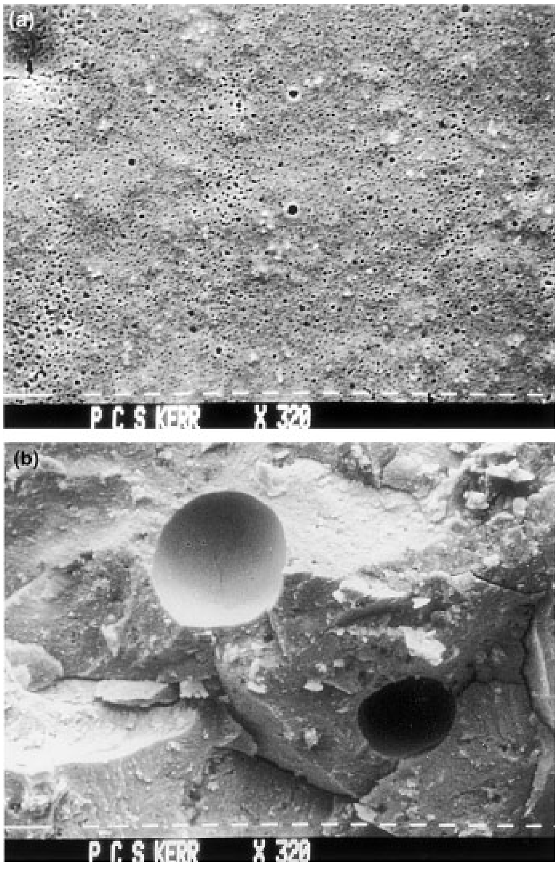

Figure 1 Pulp canal sealer cement: (a) external surface: exhibits abundant pores with diameters that do not exceed 5 μm (original magnification ×320); (b) fracture surface: smooth, with abundant vacuoles with diameters that might reach 120 μm (original magnification ×320).

4. *AH 26*: ES: markedly smooth. Scattered granules emerging towards the surface. Exceptional pores, approximately 5 μm in diameter. FS: compact and granular. Frequent vacuoles, up to 80 μm in diameter.

5. *AH Plus*: ES: smooth and granular. Exceptional pores, approximately 5 μm in diameter. FS: granular appearance. The scarce vacuoles did not exceed 80 μm in diameter, but small vacuoles, approximately 5 μm in diameter, can be observed. (Fig. 3a,b).

6. *Ketac-Endo*. ES: smooth. The surface appeared to be covered by gritty, sand-like particles. Exceptional pores, approximately 10 μm in diameter. FS: very irregular, with abundant vacuoles, homogenous in size, up to 100 μm in diameter.

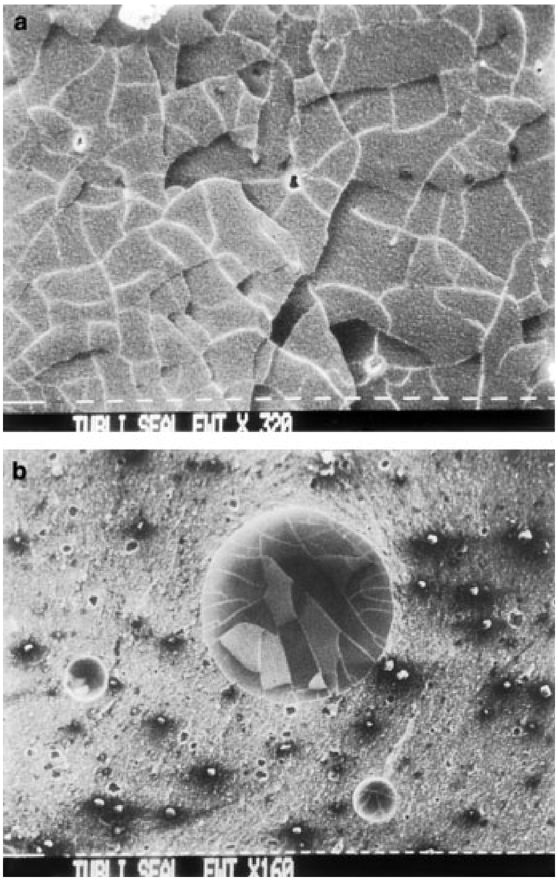

Figure 2 Tubli-seal EWT: (a) external surface: with cracks and scare pores with diameters of approximately 5 μm (original magnification ×320); (b) fracture surface: smooth, with abundant vacuoles, with diameters that might exceed 260 μm. The inner surface of the vacuole resembles the outer cracked surface (original magnification ×160).

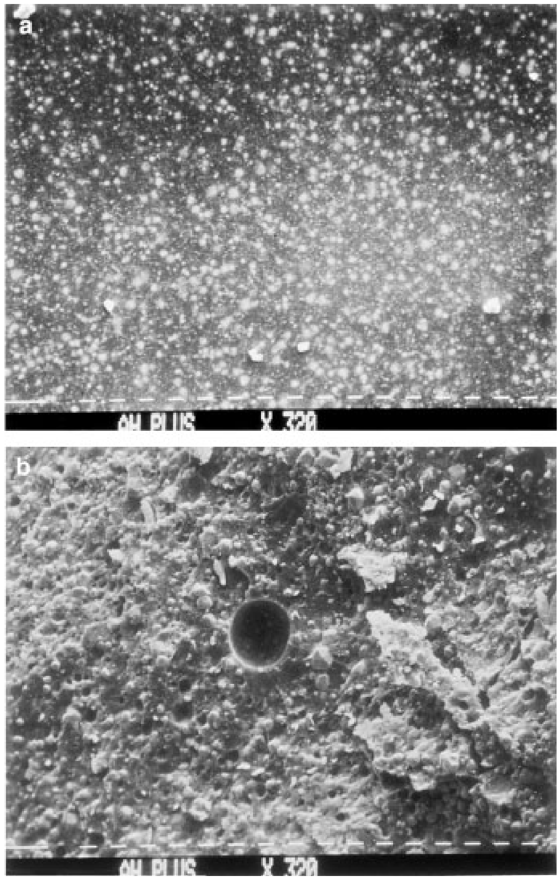

Figure 3 AH Plus: (a) external surface: smooth and granular. The area in the image fails to exhibit pores (original magnification ×320); (b) fracture surface: the scarce vacuoles can reach diameters of up to 80 μm. Small vacuoles approximately 5 μm in diameter can be observed (original magnification ×320).

7. *Apexit*. ES: abundant pores, occasionally fused, with irregular and elevated contours, up to 80 μm in diameter. The remaining surface was riddled with small pores, approximately 5 μm in diameter. FS: granular appearance. Abundant vacuoles with irregular contours, up to 120 μm in diameter.

8. *Sealer 26*. ES: abundant pores with smooth, elevated and somewhat irregular contours, up to 320 μm in diameter. Small pores were observed within. FS: slightly granular with abundant vacuoles, up to 500 μm in diameter (Fig. 4a,b).

Discussion

Roth 801 cement and Sealapex were excluded from the study; Roth 801 repeatedly failed to set (Allan *et al.*

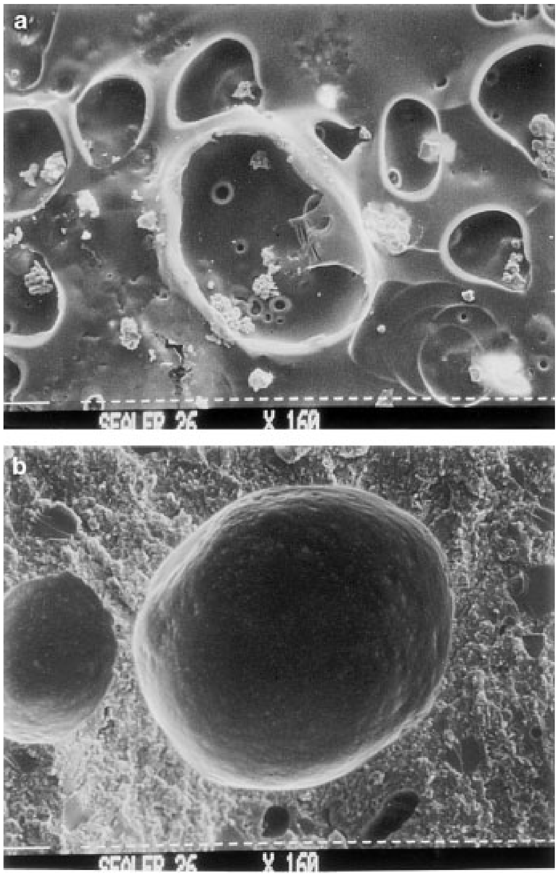

Figure 4 Sealer 26: (a) external surface: abundant pores with rounded, elevated contours and diameters that can exceed 320 μm. Pores are present on the inside (original magnification ×160); (b) fracture surface: abundant vacuoles with diameters that reach 500 μm (original magnification ×160).

2001) and Sealapex, once set, underwent excessive volumetric expansion and exhibited an elastic consistency that hindered its evaluation. These findings are in keeping with those of Caicedo & von Fraunhofer (1988).

Wesselink (1990) reported that the solid nucleus of gutta-percha of the root filling should be maximized and the amount of sealer minimized. However, this is not always possible because successful condensation of gutta-percha depends on canal morphology, the instrumentation technique employed, the shape and calibre of the gutta-percha cones and the type of spreader employed (Gani *et al.* 2000). Moreover, voids occur more frequently than expected (Manogue *et al.* 1994, Eguchi *et al.* 1985). All these factors result in an irregular distribution of the sealer. Occasionally it forms a thin layer, but frequently it forms a thick layer and might even fill large portions of the canal. It might thus become the main component of the filling. Moreover, it might even be completely missing in places (Gani *et al.* 2001).

Pores and vacuoles in sealers seem to be a consistent structural features. They were detected in varying degrees in all the sealers examined in the present as well as in previous studies (Mutal 1998, Mutal & Gani 2000). In all cases the size of the vacuoles exceeded that of pores.

Undoubtedly, both structural deficiencies originated from the air that was trapped in the sealer mass during mixing and, possibly, when it was transferred to the ring. If the air remains in the sealer as bubbles, it will give rise to vacuoles, but if it moves to the surface it will form pores. The surface of some of the sealers that exhibited only minor porosity showed marks in the shape of concentric rings. These rings would correspond to bubbles that burst open leaving faint marks. However, some bubbles might have disappeared without leaving a trace.

Evidently, the presence of these structural deficiencies depends on the physical properties of the sealer. If the mixture is dense and barely flows, only a few bubbles will open up at the interface, yielding depressions with elevated borders that resemble craters. Conversely, if the mixture is fluid, the bubbles will burst open on the surface more easily. In this case they might or might not leave traces. The fact that the vacuoles detected in zinc-eugenol-based sealers are larger than those found in resins and the glass–ionomer could be because of this phenomenon.

All the sealers under study exhibited pores and vacuoles. However, these structural defects were more frequent and larger in zinc oxide-eugenol (ZOE)-based sealers. The diameter of the vacuoles in these compounds ranged from 120 to 260 μm whereas the diameter of the vacuoles in epoxy-resins and glass–ionomer sealers did not exceed 80 μm. This finding might be attributed to differences in the consistency of the material, i.e. viscous in ZOE sealers and more flowing in epoxy-resins and glass–ionomer sealers. The flowing mixtures would allow the bubbles, in particular the larger ones, to open up to the exterior. Strangely, Sealer 26, an epoxy-resin, exhibited abundant, excessively large pores and vacuoles, 320 and 500 μm in diameter, respectively. This might be because of its calcium hydroxide content; Kazemi *et al.* (1993) reported that the OH^{-} and CA^{2+} ions in sealers dissolved leaving voids. Moreover, this process might have been enhanced by the humidity during setting.

Apexit, another sealer that contains calcium hydroxide, exhibited similar structural characteristics. The structural defects were also abundant but considerably smaller (pores: 80 μm in diameter; vacuoles: 120 μm in diameter). These characteristics might explain the solubility of this material (Rothier *et al.* 1987, Tagger & Tagger 1988), in keeping with other short-term (10 days) and mid-term studies (30 days) (Mutal 1998).

Ketac-Endo differed from the other sealers in terms of its preparation procedure. It is commercially available as capsules and is mixed using vibration without the sealer being in contact with the environment. Thus, the air within the capsule would give rise to the abundant vacuoles, up to 100 μm in diameter.

Within this context, the question arises as to the fate of the vacuoles within the sealers when the filling components are compressed within the canal. The pressure of spreaders and pluggers will probably expel the air. The air might also be trapped within the thickness of the sealer layer, however thin, not as bubbles but as small voids compressed between the gutta-percha cones or between the cones and the dentine wall.

It is thus reasonable to assume that these structural defects detected in set endodontic sealers (Mutal & Gani 2000) and their solubility (Peters 1986, Wu *et al.* 1994, 1995, Kaplan *et al.* 1997) might affect the integrity, stability, durability and the impermeability of the sealer, a pivotal component of root canal filling.

Conclusions

1. The endodontic sealers under study exhibited varying shapes and sizes of pores and vacuoles.
2. The pores and vacuoles were more frequent and larger in ZOE-based sealers than in the resins and glass–ionomer sealers. However, pores and vacuoles were more abundant in sealers containing calcium hydroxide.

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