BASIC RESEARCH - TECHNOLOGY

Volume of Voids in Retrograde Filling: Comparison between Calcium Silicate Cement Alone and Combined with a Calcium Silicate—based Sealer



Jaeheon Jung, DDS,* Sunil Kim, DDS, MSD, PhD,† Euiseong Kim, DDS, MSD, PhD,†‡ and Su-Jung Shin, DDS, MSD, PhD*

ABSTRACT

Introduction: The aim of this study was to compare the volume of voids between retrograde filling using calcium silicate cement alone and retrograde filling using a combination of calcium silicate cement with a calcium silicate-based sealer. Methods: Twenty single-rooted, extracted human teeth were instrumented with nickel-titanium files and obturated with guttapercha. We resected the roots at a point 3 mm from the apex, prepared the root ends, and filled the root-end cavities with Endocem Zr (Maruchi, Wonju, Korea) or Endoseal MTA (Maruchi) plus Endocem Zr. Then, we scanned the prepared samples using micro-computed tomographic imaging and performed 3-dimensional reconstruction. The percentage volume of the gap between the canal wall and root-end filling was calculated along with the percentage volume of voids in the filling materials. All data were analyzed using the Mann-Whitney U test. Selected specimens were further observed using scanning electron microscopy. **Results:** There were no significant differences in the percentage volumes of the gaps and internal voids between the 2 groups. Both calcium silicate and the calcium silicate-based sealer exhibited good adaptation to the cavity wall. Conclusions: The findings of our microcomputed tomographic and scanning electron microscopic analyses suggested that the volume of voids in retrograde filling using a combination of calcium silicate cement and calcium silicate-based sealer was comparable with that in retrograde filling using calcium silicate cement only. Calcium silicate-based sealers could be used in retrograde filling for clinical convenience without harmful effects on the quality of filling. (J Endod 2020;46:97–102.)

KEY WORDS

Calcium silicate; micro-computed tomography; retrograde filling; scanning electron micro-scopy; sealer

In surgical endodontic treatment, not only surgical removal of the infected root canal but also prevention of the spread of any remaining bacteria from the root canal system into the periapical tissues is essential. Therefore, a hermetic seal between the retrograde filling material and the root canal wall is an important factor that affects treatment outcomes ^{1–3}. Since its introduction in the 1990s, mineral trioxide aggregate (MTA) has emerged as the gold standard retrograde filling material for use in apical surgeries because of its superior sealing ability, biocompatibility, and capacity to induce hard tissue formation ^{4–8}. However, MTA has certain disadvantages, including a long setting time and heavy metal content. To overcome these problems, various calcium silicate cements have been introduced and widely used as retrograde filling materials ^{9–14}.

Another potential problem associated with the use of MTA is its handling difficulty. Despite the development of new calcium silicate cements and instruments, limited access and visibility during apicoectomy can result in unexpected voids in retrograde filling, which can affect treatment outcomes ¹⁵. Furthermore, the presence of anatomic complexities such as an isthmus can lower the success rate of apical surgery because of the associated difficulties in adequate retrograde preparation and filling ¹⁶. In cases of intentional replantation, although access and vision limitations are less of an issue, clinicians should finish the procedure as soon as possible to obtain a favorable prognosis. Under these conditions,

SIGNIFICANCE

The quality of retrograde fillings composed of a combination of calcium silicate cement and calcium silicate—based sealer is comparable with the quality of fillings composed of calcium silicate cement only and can be used in retrograde filling for the clinical convenience.

From the *Department of Conservative Dentistry, Gangnam Severance Dental Hospital, Yonsei University College of Dentistry, Seoul, Korea; †Microscope Center, Department of Conservative Dentistry and Oral Science Research Center, Yonsei University College of Dentistry, Seoul, Korea; and †Department of Electrical and Electronic Engineering, Yonsei University College of Engineering, Seoul, Korea

Address requests for reprints to Dr Su-Jung Shin, Department of Conservative Dentistry, Gangnam Severance Dental Hospital, Yonsei University College of Dentistry, 211 Eonjuro, Gangnam-gu, Seoul 06273, Korea. E-mail address: sujungshin@yuhs.ac 0099-2399/\$ - see front matter

Copyright © 2019 American Association of Endodontists.
https://doi.org/10.1016/
j.joen.2019.10.015

clinicians are pressed for time, which can result in low-quality retrograde filling and potential treatment failure¹⁷.

The recently developed Endoseal MTA (Maruchi, Wonju, Korea) is an injectable, premixed, bioceramic-based endodontic sealer. Because of its good flowability and handling convenience, it can be applied as a sealer with the single-cone technique used for nonsurgical endodontic treatment, with a filling quality similar to that achieved with the continuous wave technique ^{18,19}. In an *in vitro* study using an open apex model, Tran et al ²⁰ showed that the application of a calcium silicate—based sealer to the canal walls before orthograde delivery of calcium silicate—based cement enhanced its adaptation to the root dentin wall²⁰.

In apical surgery, if a calcium silicate—based sealer with good flowability is adapted to the root-end cavity before filling with conventional calcium silicate cement, it would be reasonable to expect easier manipulation, a reduced procedure time, and an improvement in the retrograde filling quality. However, studies on calcium silicate—based sealers have been limited to conventional nonsurgical root canal treatments. Therefore, the aim of this

study was to compare the volume of voids between retrograde filling using calcium silicate cement alone and retrograde filling using a combination of calcium silicate cement with a calcium silicate—based sealer.

MATERIALS AND METHODS

Sample Preparation

We collected 20 single-rooted human maxillary premolars extracted for the purpose of orthodontic treatment. The study protocol was approved by the institutional review board of our institution (no. 3-2019-0022). Teeth with 2 canals confirmed on periapical radiographs were included, whereas those with previous root canal treatment, caries, cracks, fractures, and/or perforations were excluded. A single operator prepared all specimens (J.J.), and a skilled dental hygienist assisted with material manipulation because of the fast setting time. The working length was determined at a point 0.5 mm short of the apical foramen, and the crown-down technique was selected to instrument all canals with the ProTaper Gold nickel-titanium system (Dentsply Maillefer, Ballaigues, Switzerland) up to size 40 (F4). Between instrumentations, the operator

irrigated each canal with 2.5% sodium hypochlorite solution. After final irrigation with EDTA, the canals were obturated with guttapercha cones and AH Plus sealer (Dentsply Maillefer). Subsequently, the roots were resected at a point 3 mm from the apex, 90° to the longitudinal axis, using a tapered diamond bur. Root-end preparation was performed 3 mm into the root canal space using a KiS-1D ultrasonic tip (Obtura Spartan, Algonquin, IL). To ensure uniformity of the root-end preparations, the operator made every effort to insert the tips approximately 3 mm into the canals and ensured a preparation diameter of 1.0 mm. Then, we randomly assigned the teeth to 2 groups (n = 10 each) according to the material used for retrograde filling.

Retrograde Filling Methods

In the cement group, the root-end cavities were filled with Endocem Zr (Maruchi). After mixing the material according to the manufacturer's instructions, the operator delivered it into the root-end cavity using a mineral trioxide aggregate carrier block (B&L Biotech, Ansan, Korea) and applicator (B&L Biotech). The material was packed in

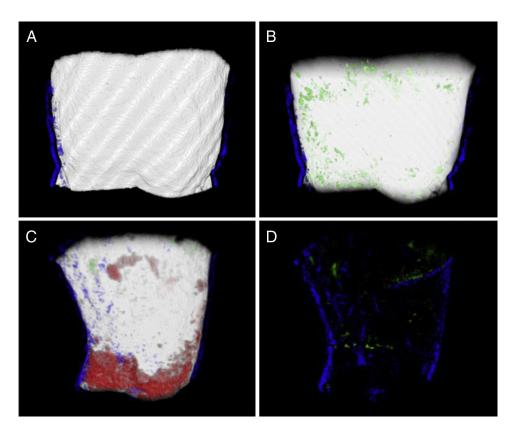


FIGURE 1 — Representative 3-dimensional micro-CT images of root canals with retrograde filling composed of calcium silicate cement alone or combined with a calcium silicate—based sealer. The *white portion* is the calcium silicate cement, and the *red portion* is the calcium silicate—based sealer. The *blue dots* represent the gap between the root-end filling materials and the tooth structure, whereas the *green dots* represent the internal voids in the root-end filling materials. (*A* and *B*) The cement group: the image (*B*) has been acquired with the transparency of the filling materials set at 50%. (*C* and *D*) The sealer + cement group: the image (*D*) has been acquired with the transparency of the filling materials set at 100%.

TABLE 1 - The Percentage Volumes for the Gap between the Retrograde Filling and the Canal Wall ((W_{out})), Internal Voids ((W_{in})), and Overall Defects ((W_{total})) in Canals with Retrograde Fillings Composed of Calcium Silicate Cement Alone or Combined with a Calcium Silicate—based Sealer (n = 10 Each)

	Group 1 (cement)	Group 2 (sealer + cement)	
Variables	Median (Q1-Q3), (minimum-maximum)	Median (Q1-Q3), (minimum-maximum)	P value
Gap (%Vout)	1.20 (0.68–5.30), (0.19–6.77)	1.37 (0.66–2.52), (0.63–4.47)	.9097
Internal voids (%V _{in})	0.13 (0.04–0.67), (0.00–0.96)	0.19 (0.02–0.36), (0.01–0.70)	.7337
Overall defects (%V _{total})	1.72 (0.87–5.30), (0.61–6.80)	1.76 (0.99–2.66), (0.68–4.52)	.7913

increments using a microplugger (B&L Biotech).

In the sealer + cement group, the rootend cavities were first filled with Endoseal MTA up to a thickness of approximately 1 mm. The operator connected a 24-G needle tip provided by the manufacturer to a syringe containing the material, carefully placed the tip in contact with the root-end cavity floor (guttapercha filling), and slowly injected the sealer. After confirming adequate coating of the rootend cavity floor using a microscope, the operator filled the rest of the cavity with Endocem Zr using the same technique described for the cement group.

All procedures were performed under an operating microscope (OPMI PICO; Carl Zeiss, Gottingen, Germany) at 10× magnification. After the retrograde filling procedure, periapical radiographs of all samples were taken to ensure the quality of

retrograde filling. All samples were stored in a humidified chamber with a relative humidity of 100% and temperature of 37° for 7 days; afterward, they were subjected to microcomputed tomographic (micro-CT) scanning.

Micro-CT Evaluation

To evaluate the amount of voids in the root-end area, we scanned all specimens using a high-resolution micro-CT scanner (SkyScan1173; Bruker, Kontich, Belgium) with an X-ray source voltage of 130 kV, a source current of 60 μ A, a pixel size of 12.04 μ m, a 1-mm-thick Al filter, an exposure time of 500 milliseconds, and a rotation step of 0.3°. Then, we used 2 software programs (Nrecon v1.7.0.4. and CTAn, Bruker) to reconstruct the raw micro-CT images and to measure the volume of the gap between the filling and root canal wall as well as the voids within the filling. Three-dimensional images of the filling were

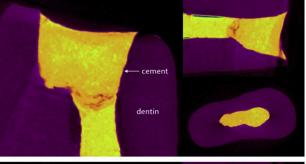
visualized using surface-rendering software (CTVox, Bruker) (Fig. 1A-D).

We included both canals of each tooth in the analysis. Measurements were performed for a 2-mm area extending apically from the interface between the gutta-percha and the root-end filling materials. In accordance with a method described in a previous study²¹, we measured the volume of the gap between the tooth surface and the retrograde filling as well as the volume of voids in the retrograde filling. A gray scale ranging from 56-255 indicated the volume of the filling (V_m), 0-10 indicated the volume of the gap between the filling and the root canal wall (Vout), and 0-56 indicated the volume of voids within the filling (V_{in}). By analyzing successive micro-CT images of each tooth, we made grayscale adjustments as needed to permit the correction of possible errors. Then, we calculated the percentage volumes using the following formulae:

$$V_{\text{out}} = V_{\text{out}}/(V_{\text{out}} + V_{\text{in}} + V_{\text{m}}) \times 100$$

$$%V_{\text{in}} = V_{\text{in}}/(V_{\text{out}} + V_{\text{in}} + V_{\text{m}}) \times 100$$

$$\%V_{\text{total}} = V_{\text{out}} + V_{\text{in}}/(V_{\text{out}} + V_{\text{in}} + V_{\text{m}}) \times 100$$



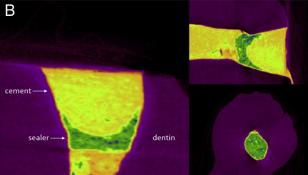


FIGURE 2 — Representative micro-CT images of a specimen from each group. The yellow portion in the root-end cavity is the calcium silicate cement, and the *green portion* in the root-end cavity is the calcium silicate—based sealer. (A) The cement group and (B) the sealer + cement group.

Scanning Electron Microscopic Analysis

After the micro-CT procedure, we randomly selected 3 teeth from each group for scanning electron microscopic analysis. The roots were embedded in light-curing embedding resin (Technovit 7200 VLC; Kulzer, Hanau, Germany) and longitudinally sectioned in the buccolingual direction using a low-speed diamond wheel (DK-2610; Struers Minitom, Rødovre, Denmark) under water cooling. The sections were dehydrated in a graded series of aqueous ethanol (30%, 50%, 70%, 90%, and 100%), vacuum dried, sputter coated with gold, and observed under a scanning electron microscope (Sigma 500; ZEISS Microscopy, Jena, Germany).

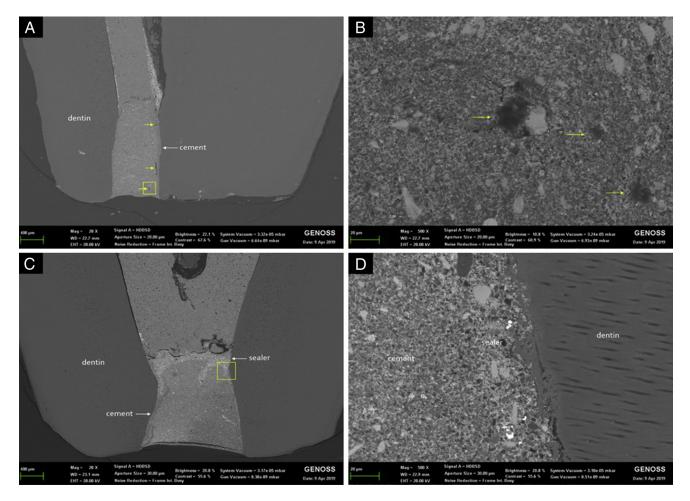


FIGURE 3 – Representative scanning electron microscopic images of root canals with retrograde fillings composed of calcium silicate cement alone or combined with a calcium silicate—based sealer. (A) The cement group (\times 20): calcium silicate cement is packed well in the root-end cavity, although some internal voids are visible (arrows). (B) The cement group (\times 500): this is a high-magnification image of the area within the *square* in A. Some internal voids (arrows) are visible. (C) The sealer + cement group (\times 20): the calcium silicate—based sealer and calcium silicate cement are packed well in the root-end cavity and can be easily distinguished. (D) The sealer + cement group (\times 500): this is a high-magnification image of the area within the *square* in C. An acceptable seal has been achieved at the interface between the calcium silicate—based sealer and the dentin.

Statistical Analysis

The Shapiro-Wilk and Kolmogorov-Smirnov tests were used to verify normal distribution of data. Because the data were not normally distributed, the Mann-Whitney U test was used to assess the statistical significance of differences between the 2 groups. All statistical analyses were performed using SAS Version 9.3 (SAS Institute, Cary, NC); the significance level was set at 95%.

RESULTS

Micro-CT Evaluation

Table 1 shows the calculated percentage volumes. There were no significant differences in the percentage volume of the gap between the root-end filling and the canal wall ($\%V_{out}$), the percentage volume of internal voids ($\%V_{in}$), and the percentage volume of overall defects ($\%V_{total}$) between the 2 groups.

The micro-CT images exhibited very few voids in the calcium silicate—based sealer, with the majority of voids located in the calcium silicate cement (Figs. 1 and 2A and B).

Scanning Electron Microscopic Analysis

There was no remarkable gap between the sealer and cement. Furthermore, the sealer exhibited an acceptable seal at its dentin interface. There were some visible internal voids in the calcium silicate cement in both groups (Fig. 3A–D).

DISCUSSION

In the present study, we used micro-CT imaging and scanning electron microscopy to evaluate the volume of voids in retrograde filling using calcium silicate cement alone or in combination with a calcium silicate—based sealer. High-resolution micro-CT imaging is a

noninvasive, highly accurate tool that has been increasingly used for the 3-dimensional assessment of microstructures. In addition to volume measurement, micro-CT facilitates qualitative analyses of images and differentiates between filling materials, voids, and tooth structures on the basis of grayscale values^{18,19,22–25}. Therefore, we used micro-CT imaging to measure the volume of the canal wall–retrograde filling gap and voids within the retrograde filling.

Unlike a previous study²¹ that only measured the volume of the gap between the tooth structure and root-end filling material, the present study additionally measured the volume of voids within the filling materials. Because a hermetic seal between the retrograde filling material and root canal wall is crucial¹⁻³, it is assumed that the gap volume directly affects retrograde filling quality, whereas the volume of internal voids may have a lesser effect on the quality¹⁹. However, their

actual relationship with apical leakage or the treatment outcomes remains unclear. In the present study, the calcium silicate—based sealer was applied in the upper part of the root-end cavity; consequently, only the upper 2-mm layer was appropriate for adequate intergroup comparisons. Therefore, we performed volume measurements for a 2-mm area extending apically from the interface between the gutta-percha and the retrograde filling.

We found that the volumes of the gap and internal voids in the sealer + cement group were smaller than those in the cement group, although there was no statistically significant difference between the 2 groups. Furthermore, the volumes in the former group exhibited a narrower range than did those in the latter group. These results imply that retrograde filling with a combination of calcium silicate cement and calcium silicate—based sealer is a less technique-sensitive procedure.

We used scanning electron microscopy to observe the interface between the retrograde filling and the root dentin in the 2 groups. However, scanning electron microscopy provides 2-dimensional images; moreover, there is a possibility of detachment of the filling material from the root dentin under the high-vacuum setting. Nevertheless, scanning electron microscopy is an effective method for the assessment of marginal adaptation because of its good resolution and high magnification^{26,27}. Furthermore, in this study, selected teeth for scanning electron microscopic analysis were embedded in

embedding resin to decrease the possibility of cracking or detachment during specimen preparation. Our scanning electron microscopic analyses showed that the quality of retrograde filling composed of a combination of calcium silicate-based sealer and calcium silicate cement was acceptable, with no significant gaps and/or voids at the interface. This may be attributed to the presence of similar components in the 2 materials. Endocem Zr is derived from pozzolan cement and is composed of calcium oxide, silicon dioxide, aluminum oxide, zirconium oxide, and other metallic oxides²⁸. Endoseal MTA has been developed by the same company and is also based on pozzolan cement. High-magnification scanning electron microscopic images obtained in the present study showed that Endoseal MTA achieved an excellent seal at its interface with the tooth structure. Furthermore, Yoo et al²⁹ detected mineralized apatite structures throughout the dentinal tubules in canals obturated with Endoseal MTA. The authors speculated that the very fine texture (mean particle size = 1.5 μm) of the material contributed to this phenomenon²⁸. This biomineralization ability may have contributed to the superior sealing morphology at the interface between Endoseal MTA and the tooth structure in the present

This study has some limitations. First, it was an *ex vivo* study, and all procedures were performed in an environment similar to that during intentional replantation. If the same procedure had been performed under

conditions mimicking those during apicoectomy, with dentiform teeth fixed in a phantom, the results may have been somewhat different. Furthermore, in this study, we included maxillary premolars that had 2 canals to better reflect the clinical difficulties associated with anatomic complexities such as an isthmus, but the difficulty level during actual clinical procedures varies according to tooth type, tooth position, number of roots, and root form. Accordingly, further studies following consistent guidelines for this procedure are necessary.

In conclusion, within the study limitations, the findings of our micro-CT imaging and scanning electron microscopic analyses suggested that the volume of voids in the retrograde filling composed of a combination of calcium silicate cement and calcium silicate—based sealer was comparable with that in the retrograde filling composed of calcium silicate cement only. These findings are clinically relevant because they suggest calcium silicate—based sealer could be used in retrograde filling for clinical convenience without harmful effects on the quality of filling.

ACKNOWLEDGMENTS

Supported by the National Research Foundation of Korea funded by the Korean government (Ministry of Science, ICT and Future Planning) (grant no. 2017R1A2B1008405).

The authors deny any conflicts of interest related to this study.

REFERENCES

- Christiansen R, Kirkevang LL, Hørsted-Bindslev P, et al. Randomized clinical trial of root-end resection followed by root-end filling with mineral trioxide aggregate or smoothing of the orthograde gutta-percha root filling—1-year follow-up. Int Endod J 2009;42:105–14.
- Kruse C, Spin-Neto R, Christiansen R, et al. Periapical bone healing after apicoectomy with and without retrograde root filling with mineral trioxide aggregate: a 6-year follow-up of a randomized controlled trial. J Endod 2016;42:533–7.
- 3. Mente J, Leo M, Michel A, et al. Outcome of orthograde retreatment after failed apicoectomy: use of a mineral trioxide aggregate apical plug. J Endod 2015;41:613–20.
- 4. von Arx T, Hänni S, Jensen SS. 5-year results comparing mineral trioxide aggregate and adhesive resin composite for root-end sealing in apical surgery. J Endod 2014;40:1077–81.
- Tang Y, Li X, Yin S. Outcomes of MTA as root-end filling in endodontic surgery: a systematic review. Quintessence Int 2010;41:557–66.
- 6. Çalışkan MK, Tekin U, Kaval ME, et al. The outcome of apical microsurgery using MTA as the rootend filling material: 2- to 6-year follow-up study. Int Endod J 2016;49:245–54.
- Kim E, Song JS, Jung IY, et al. Prospective clinical study evaluating endodontic microsurgery outcomes for cases with lesions of endodontic origin compared with cases with lesions of combined periodontal-endodontic origin. J Endod 2008;34:546–51.
- Song M, Kim E. A prospective randomized controlled study of mineral trioxide aggregate and super ethoxybenzoic acid as root-end filling materials in endodontic microsurgery. J Endod 2012;38:875–9.

- 9. Asgary S, Ehsani S. Periradicular surgery of human permanent teeth with calcium-enriched mixture cement. Iran Endod J 2013;8:140–4.
- Shinbori N, Grama AM, Patel Y, et al. Clinical outcome of endodontic microsurgery that uses EndoSequence BC root repair material as the root-end filling material. J Endod 2015;41:607–12.
- Zhou W, Zheng Q, Tan X, et al. Comparison of mineral trioxide aggregate and iRoot BP Plus Root Repair Material as root-end filling materials in endodontic microsurgery: a prospective randomized controlled study. J Endod 2017;43:1–6.
- 12. Biočanin V, Antonijević Đ, Poštić S, et al. Marginal gaps between 2 calcium silicate and glass ionomer cements and apical root dentin. J Endod 2018;44:816–21.
- 13. Antunes HS, Gominho LF, Andrade-Junior CV, et al. Sealing ability of two root-end filling materials in a bacterial nutrient leakage model. Int Endod J 2016;49:960–5.
- Küçükkaya S, Görduysus MÖ, Zeybek ND, et al. In vitro cytotoxicity of calcium silicate based endodontic cement as root-end filling materials. Scientifica (Cairo) 2016;2016:9203932.
- Villa-Machado PA, Botero-Ramírez X, Tobón-Arroyave SI. Retrospective follow-up assessment of prognostic variables associated with the outcome of periradicular surgery. Int Endod J 2013;46:1063–76.
- Kim S, Jung H, Kim S, et al. The influence of an isthmus on the outcomes of surgically treated molars: a retrospective study. J Endod 2016;42:1029–34.
- 17. Jang Y, Lee SJ, Yoon TC, et al. Survival rate of teeth with a C-shaped canal after intentional replantation: a study of 41 cases for up to 11 years. J Endod 2016;42:1320–5.
- Kim SR, Kwak SW, Lee JK, et al. Efficacy and retrievability of root canal filling using calcium silicate-based and epoxy resin-based root canal sealers with matched obturation techniques. Aust Endod J 2019. https://doi.org/10.1111/aej.12323 [Epub ahead of print].
- Kim S, Park JW, Jung IY, et al. Comparison of the percentage of voids in the canal filling of a calcium silicate-based sealer and gutta percha cones using two obturation techniques. Materials (Basel) 2017;10.
- Tran D, He J, Glickman GN, et al. Comparative analysis of calcium silicate-based root filling materials using an open apex model. J Endod 2016;42:654–8.
- 21. Kim SY, Kim HC, Shin SJ, et al. Comparison of gap volume after retrofilling using 4 different filling materials: evaluation by micro-computed tomography. J Endod 2018;44:635–8.
- 22. El-Ma'aita AM, Qualtrough AJ, Watts DC. A micro-computed tomography evaluation of mineral trioxide aggregate root canal fillings. J Endod 2012;38:670–2.
- 23. Hammad M, Qualtrough A, Silikas N. Evaluation of root canal obturation: a three-dimensional in vitro study. J Endod 2009;35:541–4.
- 24. Jung M, Lommel D, Klimek J. The imaging of root canal obturation using micro-CT. Int Endod J 2005;38:617–26.
- 25. Wolf M, Küpper K, Reimann S, et al. 3D analysis of interface voids in root canals filled with different sealer materials in combination with warm gutta-percha technique. Clin Oral Investig 2014:18:155–61.
- Tanzilli JP, Raphael D, Moodnik RM. A comparison of the marginal adaptation of retrograde techniques; a scanning electron microscopic study. Oral Surg 1980;50:74–80.
- 27. Stabholz A, Friedman S, Abed J, et al. Marginal adaptation of retrograde fillings and its correlation with sealability. J Endod 1985;11:218–23.
- 28. Choi Y, Park SJ, Lee SH, et al. Biological effects and washout resistance of a newly developed fast-setting pozzolan cement. J Endod 2013;39:467–72.
- Yoo YJ, Baek SH, Kum KY, et al. Dynamic intratubular biomineralization following root canal obturation with pozzolan-based mineral trioxide aggregate sealer cement. Scanning 2016;38:50–6.