

# Marginal Gaps between 2 Calcium Silicate and Glass Ionomer Cements and Apical Root Dentin

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## Abstract

**Introduction:** The outcome of periapical surgery has been directly improved with the introduction of novel material formulations. The aim of the study was to compare the retrograde obturation quality of the following materials: calcium silicate (Biodentine; Septodont, Saint-Maur-des-Fosses, France), mineral trioxide aggregate (MTA+; Cerkamed Company, Stalowa Wola, Poland), and glass ionomer cement (Fuji IX; GC Corporation, Tokyo, Japan). **Methods:** Materials' wettability was calculated concerning the contact angles of the cements measured using a glycerol drop. Cements' porosity was determined using mercury intrusion porosimetry and micro-computed tomographic ( $\mu$ CT) imaging. Extracted upper human incisors were retrofilled, and  $\mu$ CT analysis was applied to calculate the volume of the gap between the retrograde filling material and root canal dentin. Experiments were performed before and after soaking the materials in simulated body fluid (SBF). **Results:** No statistically significant differences were found among the contact angles of the studied materials after being soaked in SBF. The material with the lowest nanoporosity (Fuji IX: 2.99% and 4.17% before and after SBF, respectively) showed the highest values of microporosity (4.2% and 3.1% before and after SBF, respectively). Biodentine had the lowest value of microporosity (1.2% and 0.8% before and after SBF, respectively) and the lowest value of microgap to the root canal wall ( $[10 \pm 30] \times 10^{-3} \text{ mm}^3$ ). **Conclusions:** Biodentine and MTA possess certain advantages over Fuji IX for hermetic obturation of retrograde root canals. Biodentine shows a tendency toward the lowest marginal gap at the cement-to-dentin interface. (*J Endod* 2017; ■:1–6)

## Key Words

Calcium silicate, micro-computed tomographic imaging, porosity, root-end filling, wettability

The main goal of periapical surgery is to remove pathological periradicular tissue and hermetically seal the root apex in 3 dimensions (1). The literature confirms that if an optimal apical seal is not achieved, *restitutio ad integrum* may not occur (2–4). The outcome of periapical surgery has been directly improved with the introduction of tricalcium silicate material formulations (5–8).

Mineral trioxide aggregate (MTA) has biocompatibility, radiopacity, and superior obturation quality compared with amalgam, Super EBA (Bosworth, Chicago, IL), and intermediate restorative material cement (9, 10). MTA possesses adequate sealing ability preventing microbial contamination and its by-products (11–13). However, MTA has a long setting time and difficulties with handling and compacting into narrow and curved root canals. Biodentine (BD; Septodont, Saint-Maur-des-Fosses, France) is a novel formulation of tricalcium silicate-based cement designed to overcome these drawbacks. It presents excellent 3-dimensional (3D) apical obturation followed by the lowest level of microleakage in comparison with glass ionomers (GIs) and MTA (14).

From a clinical point of view, hermetic obturation is directly correlated to bacterial leakage and, consequently, contamination of periapical tissues. First, bacterial leakage is significantly reduced if root-end filling material possesses low porosity (15). Porosity has a direct effect on the permeability of bacteria and their products into periapical tissues (16). Second, surface features of root-end filling material are related to its potential to adhere to surrounding dental tissue. High wettability of root-end filling materials is desired because a positive correlation has been found with the sealing ability and material penetration into dentinal tubules (17).

The aim of this *in vitro* study was to evaluate the quality of 3D retrograde filling by comparing the wettability, the porosity of selected materials per se in clinically simulated conditions, and the volume of the gap at the material-dentin interface among GI and 2

## Significance

Adequate 3D obturation is a crucial prerequisite for the prevention of the postoperative spread of periapical infection. Here, we document that Biodentine presents certain microstructural advantages over MTA+ and Fuji IX in retrograde root canal surgery.

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## Basic Research—Technology

tricalcium silicate–based cements. The null hypothesis was that the choice of the material has no effect on the quality of 3D retrograde obturation.

### Materials and Methods

#### Specimen Preparation

Fifteen human, single-rooted, upper, frontal teeth extracted for periodontal reasons were used in this study. Teeth were free of root caries, fractures, resorption, calcifications, curvature of the root canal, and previous endodontic treatment. All teeth were disinfected by immersion in 5.25% sodium hypochlorite for 6 hours and stored in sterile saline. Pulp was extirpated in all teeth with a #15 K-Flex file (Kerr Corporation, Orange, CA), and the canals were prepared with the step back technique using 3% sodium hypochlorite as irrigant and dried with absorbent paper points. The canals were enlarged up to a #40 K-File (Kerr Corporation) size. Root canals were obturated by the lateral compaction technique using gutta-percha (Hygenic; Coltène/Whaledent AG, Altstätten, Switzerland) and AH Plus sealer (Dentsply De Trey GmbH, Konstanz, Germany). Root ends were resected 3 mm from the apex at 90° to the long axis of the root. Resection was performed using a diamond fissure bur and irrigation with saline. A 3-mm-deep and 3- to 4-mm-wide (depending on root-end anatomy) retrograde preparation was performed with ultrasonic retrotips. Teeth were randomly divided into 3 groups ( $n = 5$  each), and retrograde cavities were filled with BD (tricalcium silicate with zirconium oxide [BD group]), GI (Fuji IX GP [GI group]), or MTA (calcium silicate with bismuth oxide [MTA group]) (MTA+; Cerkamed Company, Stalowa Wola, Poland) mixed according to the manufacturers' instructions. Retrocavities for GI were conditioned with polyacrylic acid before its placement. Digital retroalveolar radiographs were taken to confirm the quality of obturation (Trophy Radiology; Cedex, Saint Maur, France). All measurements were performed before and after soaking specimens in simulated body fluid (SBF) for 4 weeks.

#### Wettability Measurements

Glycerol solution was chosen as the reference liquid for static contact angle (CA) measurements in accordance with previous studies that proved it to be an ideal blood analog that falls within the normal range of human blood viscosity (18). The method consisted of applying a sessile drop (2  $\mu$ L) of the reference liquid with a syringe onto a specimen. All measurements were performed 10 seconds after placing the liquid droplet at the materials' surfaces when equilibrium was established at the 3-phase boundary where glycerol, surrounding air, and cement intersect. The profile of the liquid drops was recorded using a CA analyzer. The CAs were calculated using ImageJ software (National Institutes of Health, Bethesda, MD), which allowed a general conic section equation to be fit to the contour of the droplet placed on the surface (19).

#### Micro- to Nanoporosity

The microporosity of the cements was determined by scanning the specimens with a micro-computed tomographic ( $\mu$ CT) scanner (SkyScan 1172; Bruker microCT, Kontich, Belgium) operated at 73 kV and 135 mA with an exposure time of 1200 milliseconds, a copper aluminum filter, a rotation of 180° in 0.4 steps, frame averaging of 3.5- $\mu$ m isotropic resolution, and 2048  $\times$  2048 pixels per slice. The specimens were 10 mm in thickness and 6 mm in diameter. The acquired images were reconstructed using NRecon v.1.6.9.8 software (SkyScan) with a threshold of 0 to 0.065, ring artifact correction of 9%, beam hardening correction of 54%, and smoothing of 1. Images were analyzed with CTAn 1.16.4.1 software (SkyScan). The global threshold gray value was set from 90 to 255. The following parameters were calculated: total porosity (%), closed porosity (%), open porosity (%), pore diameter (mm), and the number of closed pores (1/mm<sup>3</sup>).

To measure the nanosized pores, the high-pressure mercury intrusion porosimeter (Porosimeter 2000; Carlo Erba, Milan, Italy) was used, operating in the 0.1–200 MPa interval. The Milestone 100 Software System (Milestone Systems, Brøndby Municipality, Denmark) was used to estimate the pores in an interval of 7.5–15000 nm. Nanoporosity measurements of the specimens were calculated using a pycnometer with benzene as the displacement fluid. Preparation of the samples was performed at room temperature and a pressure of 0.5 kPa.

#### Measurement of the Volume of the Gap between Cement and Root Canal Walls

The teeth were scanned using  $\mu$ CT imaging and the same scanning and reconstruction parameters as described for the cement microporosity analysis. The calculation of the volume of the gap between the cement and the root canal wall was performed using CTAn 1.16.4.1 software. The volume of interest was defined by the first 400 slides at the most apical part of the root end. The region of interest (ROI) was performed in software computed tomographic analyzer thresholding between 149 and 255 to define the region including cement material, despeckling all except the largest object in 3 dimensions, closing in 2-dimensional (2D) space at a radius of 15 pixels, dilatation in 2D space at a radius of 10 pixels, copying the image to define the first ROI, dilatation in 2D space at 20 pixels and subtracting the first ROI from the image to define the definitive ROI, reloading the image and thresholding it at a ratio between 32 and 255 to visualize the interface void, and 3D analysis to calculate the total volume of pore space and average void diameter.

#### Statistics

Statistical analysis was performed using SPSS 20.0 (SPSS Inc, Chicago, IL). For wettability measurements, differences among the tested materials were evaluated for significance by means of 1-way analysis of variance with the Tukey post hoc test, whereas within-group comparisons were performed using the paired sample *t* test. Porosity values were analyzed using the Kruskal-Wallis test among the groups followed by the Mann-Whitney *U* post hoc test with Bonferroni-Holm correction. Within groups, porosity was tested for significant differences using the Wilcoxon signed rank test. Differences were significant at a 95% confidence interval ( $P < .05$ ).

### Results

The results of the wettability measurements are shown in Figure 1. A comparison of CAs before and after submersion of the materials in SBF revealed statistically significant differences in the BD and GI groups ( $P < .05$ ). GI presented significantly higher values of CAs than BD and MTA before submersion in SBF. After submersion in SBF, BD showed significantly higher values than GI and MTA ( $P < .05$ ).

3D images of the investigated cements' microporosity obtained by  $\mu$ CT imaging are shown in Figure 2A. No statistically significant differences were measured before or after submersion of the materials in SBF in any of calculated microporosity parameters ( $P > .05$ ) (Fig. 2C). Statistical significance was found for closed porosity between MTA and BD before submersion in SBF. The number of closed pores was significantly lower in the GI group than in the BD and MTA groups before submersion in SBF. After submersion in SBF, significant differences were found in the average pore size; GI had higher porosity than BD and MTA ( $P < .05$ ), whereas BD presented significantly more closed pores than MTA ( $P < .05$ ). Regarding other parameters (Fig. 2), no statistically significant differences were measured. Higher values of total porosity were only observed in the GI group, both before and after submersion in SBF.

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