New Tetrasilicate Cements as Retrograde Filling Material: An In Vitro Study on Fluid Penetration

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Abstract

We aimed to compare the apical sealing ability of two experimental retrograde root-filling cements and mineral trioxide aggregate using a fluid filtration method. X-ray diffraction (XRD) and X-ray fluorescence spectrometry (XRF) were used to evaluate structural and qualitative characteristics. Thirty single-rooted extracted teeth were treated, root-end prepared, and obturated using MTA and two experimental cements. Fluid filtration was measured during a 5-minute period after 4, 24, and 48 hours and 1, 2, and 12 weeks. The results were statistically compared by using a two-way analysis of variance ($p < 0.05$). The marginal adaptation was evaluated by using a SEM replica technique. XRD analyses showed similar patterns. XRF showed lower amounts of SiO_2 and FeO_3 in the experimental cements. All cements showed a reduced fluid flow rate over time. No significant differences were found between the cements. The SEM replica indicated a good marginal adaptation to dentinal walls. Both experimental cements showed suitable properties as retrograde root-filling materials. *(J Endod 2007;33:742–745)*

Key Words

Endodontic cements, fluid filtration method, root-end–filling material, sealing ability tests, SEM replica, X-ray diffraction, X-ray fluorescence spectrometry

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The presence of marginal gaps between the interfaces of retrofilling materials and root dentin are sites of possible microleakage (1, 2) and passage of bacteria and their toxins (3, 4).

Mineral trioxide aggregate (MTA) has been recently introduced as retrograde filling material (5–7). MTA derives from Portland cement and contains tricalcium silicate, tricalcium aluminate, tricalcium oxide, and silicate oxide (5). Osteoblast-like cells showed similar growth and matrix formation when grown on either set MTA or Portland cement (8, 9).

Poor handling characteristic and lengthy setting time are reported by clinicians when using MTA. It would be useful to expand the applications of MTA by improving those characteristics (10). Furthermore, a recent study reported that root-end fillings performed using MTA presented microleakage (11).

The purposes of this study were to compare (1) the chemical and crystallographic properties and the pH of two experimental mineral cements, MTA and of Portland cement using X-ray diffraction (XRD) and X-ray fluorescence spectrometry (XRF); (2) the apical sealing ability of the experimental cements and MTA using a fluid filtration method; and (3) the morphology and the marginal adaptation of root canal walls of these endodontic cements using an impression replica technique and scanning electron microscopy.

Materials and Methods

Two experimental accelerated Tetrasilicate Cements (TC-1 and TC-2) were formulated. The composition was: Portland cement (CEM II/B-LL 32,5 R Aquila Bianca; Italcementi, Bergamo, Italy), 14.5% (wt) of bismuth oxide as radiopacifier, 5% (wt) of calcium chloride as accelerant, and 1% and 2% (wt) of a phyllosilicate as plasticizing agent respectively in TC-1 and TC-2. White-MTA (ProRoot Dentsply-Maillefer, Ballagues, Switzerland) was used as control material.

pH Measurement

The cements were inserted into plastic tubes (1-mm diameter and 10-mm length). Five samples were prepared for each material. Each tube was immersed in 10 mL of deionized water and stored at 37°C in a 100% relative humidity environment for 4, 24, and 48 hours. Then the tubes were placed into 10 mL of fresh deionized water. The pH of storage solution was measured by using a pH meter (Phoenix K0201B; Phoenix Electrode Company, Houston, TX).

Cements Characterization

Structural characterization (XRD) was performed by using an automated powder diffractometer (Philips PW 3719; Philips, Eindhoven, The Netherlands) with CuKα radiation and a secondary crystal monochromator. Chemical characterization (XRF) was performed by using a quantitative X-ray fluorescence spectrometer (Philips PW 1480, Philips). All the samples were analyzed in powder form.

Endodontic Sample Preparation

Thirty single-rooted teeth extracted for periodontal reasons with similar length were used. The crowns were cut at the cementoenamel junction (CEJ); the root canals were prepared with a crown-down technique. Canals instrumentation was performed to

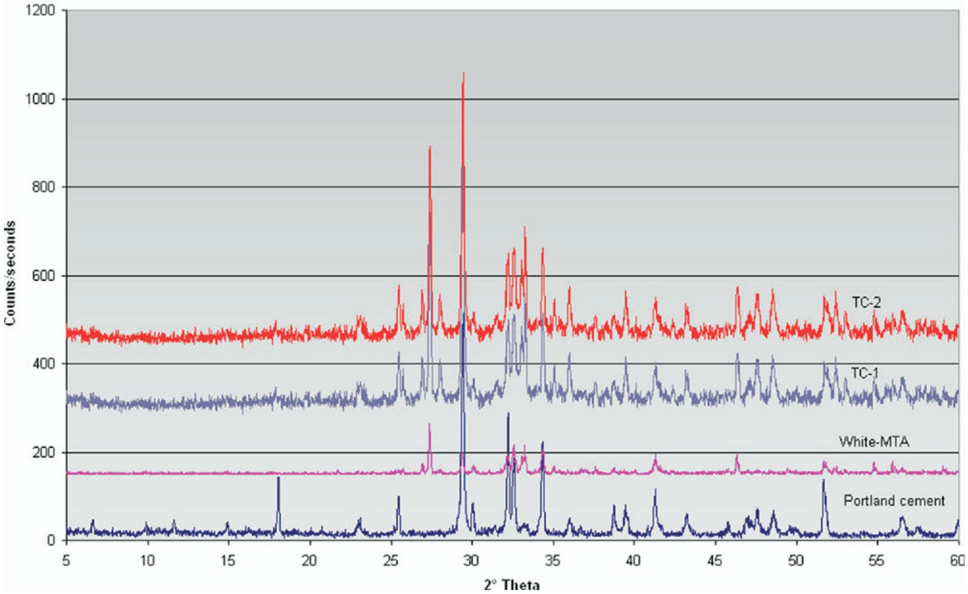

Figure 1. XRD patterns of experimental cements TC-1 and TC-2, white MTA, and Portland cement indicating the presence of the same main constituent (of same phases in the materials composition).

#30 K file about 1 mm shorter of the external apical foramen. Irrigation with 5 mL NaOCl 5% (Ogna, Muggiò-Milano, Italy) and 2 mL EDTA 17% (Ogna) was performed throughout the instrumentation. Samples were filled using gutta percha with lateral condensation technique without sealer to easily remove the gutta percha after root-end filling. Apicoectomy was performed by using a cylindrical diamond bur mounted on a high-speed hand piece at approximately 3 mm from the apex. The root samples were randomly divided in 3 groups of 10 samples each filled with the different cements. Portland cement was not tested for the sealing ability because it is not used for clinical application. A control group with no retrofilling material was not prepared because the greater filtration rate observed in preliminary tests.

Finally, the gutta percha was removed, and the external root surface was covered with nail varnish except for the apical 3 mm. During the periods between the leakage measurements, the teeth were stored in deionized water at 4°C.

Fluid Filtration Evaluation

The root samples were fixed by CEJ using cyanocrylate to plexiglass supports crossed by an 18-gauge stainless steel tube, and then they were connected to a fluid conductive system working at a hydraulic pressure of 6.9 KPa to measure the fluid movement (4). The fluid filtration rate toward the roots was measured over three 5-minute periods at 1-minute intervals and the mean calculated. The results were expressed as μL/min. After 4, 24, and 48 hours and 1, 2, and 12 weeks from root-filling treatment, the sealing ability (i.e., filtration rate) was measured.

The mean fluid filtration values were statistically analyzed by using a two-way analysis of variance test (at a significance level of $p < 0.05$). Comparisons between the mean filtration values of the different cements at each given time and between the mean filtration values of each cement at each given time were performed.

Interfacial Morphology Evaluation

A SEM replica technique ($n = 5$) was used to the interface between the retro-filling materials and the root canal walls. To avoid the artifacts that may be induced in water-containing dental materials by dehydratation required in conventional samples preparation for SEM, positive replicas were fabricated from the polyvinylsiloxane impressions using a polyether impression material (Permadyne Garant, 3M/ESPE, St. Paul, MN), according to technique reported by Chersoni et al. (12). The positive replicas were coated with gold and examined by SEM (Model 5400; JEOL, Tokyo, Japan) at 10 kV.

Results

XRD structural analyses of TC-1 and TC-2 showed their complete crystallinity, with definite peaks attributable to specific phases. The same main constituent elements were observed in white MTA diffractograms (Fig. 1). TC-1, TC-2, MTA, and Portland cement showed similar patterns. No reflex of phyllosilicate introduced to optimize the products plasticity and workability was present in the pattern of the experimental cements because its percentage rate was lower than the instrument sensitivity.

XRF quantitative analyses showed a different composition between both experimental cements, white MTA, and Portland cement. In particular, in the experimental cements lower amounts of SiO_2 and Fe_2O_3 compounds compared with MTA and Portland cement were found (Table 1).

Table 2 shows the pH measurements of MTA, Portland cement, and experimental cements. TC-1 and TC-2 showed a pH slightly inferior to

TABLE 1. Table Showing the Quantitative XRF Analysis Values for Experimental Cements, White MTA, and Portland Cement Groups

	TC-1	TC-2	White MTA	Portland Cement
SiO_2 (%)	13.87	13.93	27.31	18.76
TiO_2 (%)	0.04	0.07	0.08	0.37
Al_2O_3 (%)	2.14	2.15	2.12	5.04
Fe_2O_3 (%)	0.80	0.78	1.11	4.42
FeO (%)	0.00	0.00	0.00	0.00
MnO (%)	0.00	0.00	0.01	0.02
MgO (%)	1.15	1.18	1.10	2.16
CaO (%)	68.71	69.79	65.10	59.07
Na_2O (%)	0.51	0.55	0.37	0.27
K_2O (%)	0.18	0.17	0.16	1.01
P_2O_5 (%)	0.03	0.05	0.21	0.08
LOI (%)	12.57	12.55	2.43	8.81