Sealers and Warm Gutta-percha Obturation Techniques

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Abstract

Introduction: Warm vertically compacted gutta-percha obturation techniques use root canal sealers that are heated during the obturation. This study aims at investigating the suitability of selected sealers with warm gutta-percha obturation techniques. **Methods:** The composition of an experimental sealer (Septodont; Saint Maur-des-Fosses, France), MTA Fillapex (Angelus, Londrina, Brazil), Apexit Plus (Ivoclar, Schaan, Lichtenstein), and AH Plus (Dentsply International, Addlestone, UK) was assessed by scanning electron microscopic and energy-dispersive spectroscopic analysis. The effect of temperature during warm vertical compaction technique was investigated by testing the sealers' properties after 1 minute to 100°C or 37°C. The reaction products after setting were assessed by X-ray diffraction analysis and Fourier transform infrared spectroscopy. Changes in setting time, flow, and film thickness were determined using ISO 6876 (2012) specifications. **Results:** The experimental tricalcium silicate–based sealer and Apexit Plus contained calcium hydroxide peaks after setting, which were absent in MTA Fillapex. The properties of AH Plus and the experimental sealer were modified by heat; the setting time was reduced, and film thickness increased. AH Plus had diminished N-H groups when heated to 100°C for 1 minute. MTA Fillapex, Septodont sealer, and Apexit Plus were unaffected by heat application. **Conclusions:** The choice of sealer should be considered when selecting the obturation technique. The Septodont sealer is recommended for obturations using cold laterally condensed gutta-percha, whereas MTA Fillapex and Apexit Plus were suitable with warm gutta-percha obturation techniques. *(J Endod 2015;41:72–78)*

Key Words

AH Plus, Apexit Plus, characterization, MTA Fillapex, physical properties, root canal sealers, Septodont experimental tricalcium silicate–based sealer, warm vertical compaction

Obturation of the root canal involves the use of gutta-percha in combination with root canal sealer to provide an adequate seal. The use of sealer is necessary to fill voids and gaps between the main material and the root canal walls. Without a sealer, canal obturations exhibit greater leakage (1, 2).

Warm gutta-percha obturation techniques have been developed to produce 3-dimensional root canal obturations because thermoplasticized gutta-percha can fill better canal irregularities than solid gutta-percha points (3). The phase changes of gutta-percha as a function of temperature have been reported, and gutta-percha exhibits 2 phase changes with a rise in temperature; namely, it goes from beta to alpha phase and then amorphous and from amorphous to beta on cooling (4–6). The maximum temperature required to achieve the amorphous phase in gutta-percha is 60°C (5). Regardless of the low temperature required to cause phase changes in gutta-percha, most thermoplasticized systems operate at 200°C. The temperature at the tip of the pluggers is much lower than the temperature of 200°C set on the liquid crystal display. Previous research on different thermoplasticized gutta-percha units reported temperatures approximately 50°C below the liquid crystal display readout when settings were above 200°C (7). The highest temperature reported in 0.06 taper System B Pluggers (Sybron-Endo, Orange, CA) was 80°C at the shank, whereas lower temperatures were measured at the tip and middle part of the plugger (8).

The effect of temperatures during warm vertical compaction on root canal sealers has not been extensively investigated. The effects of temperature on AH Plus (Dentsply International, Addlestone, UK), MTA Fillapex (Angelus, Londrina, Brazil), pulp canal sealer, and a prototype resin-based material have recently been reported (8). AH Plus obturations caused higher temperatures at the external root surface, and the chemical composition of AH Plus sealer was affected by high temperature. Analysis of heated AH Plus showed variations in the high-frequency part of the infrared spectrum between 4000 and 1300/cm. The stretching vibration of the nitrogen to hydrogen bond (N-H) group present at 2900/cm was absent after heat was applied (8, 9).AH Plus contains dibenzyldiamine, aminoadamantane, and tricyclodecane-diamine in paste B. These polyamines act as initiators and react with the resins in paste A, resulting in polymerization. The heat seems to disintegrate these phases. The amines present in a prototype epoxy resin–based sealer were unaffected (9). Furthermore, a reduction in sealer setting time and strength was observed. The heat did not affect the pulp canal sealer or MTA Fillapex (8).

Investigation of MTA Plus, AH Plus, and 2 prototypes based on radiopacified tricalcium silicate using water or epoxy resin as vehicles showed that although the water-based prototype sealer and MTA Plus had a similar chemical composition, MTA Plus was unaffected by heat application as opposed to the prototype water-based sealer, which exhibited flattening out of the O-H stretching vibration at 3400/cm. The application of heat evaporated the water present in the sealer composition (9). Sealer porosity was considerably reduced in all sealer types (9).

Recently, a novel tricalcium silicate–based sealer has been introduced by Septodont (Saint Maur-des-Fosses, France). According to the manufacturer, this sealer is

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indicated for use in obturations using cold laterally condensed gutta-percha. The aim of this research was to investigate the suitability of selected sealers to be used with warm gutta-percha obturation techniques.

Materials and Methods

The following materials were used in this study:

1. MTA Fillapex
2. Experimental tricalcium silicate–based sealer (Septodont)
3. Apexit Plus (Ivoclar, Schaan, Lichtenstein)
4. AH Plus

The material composition supplied by the manufacturer is shown in Table 1. The materials were characterized by scanning electron microscopy and energy-dispersive spectroscopy (EDS) to determine the constituents, thus enabling the interpretation of the changes sustained by the material after the application of heat.

Characterization of Materials by Scanning Electron Microscopy and EDS

Cylindric specimens with a 10-mm diameter and 2-mm high were prepared. They were allowed to set for 48 hours at 37°C and 100% humidity in a climatic chamber (Weiss-Gallenkamp, Loughborough, UK). The materials were then embedded in cold cure resin (Epoxyfix; Struers GmbH, Ballerup, Denmark) and polished with progressively finer grits of diamond discs and polishing clots with diamond suspensions finishing with a silicon suspension of 1 μm using an automatic polishing machine (Tegramin 20; Struers, Ballerup, Denmark). The specimens were mounted on aluminum stubs, carbon coated, and viewed with a scanning electron microscope (Zeiss MERLIN Field Emission SEM; Carl Zeiss NTS GmbH, Oberkochen, Germany). Scanning electron micrographs were captured of the material microstructural components in the backscatter electron mode, and EDS was performed over a wide area.

Assessment of the changes in material composition (ie, physical and chemical properties) when the sealer was used during warm vertical compaction of gutta-percha was performed after subjecting the sealer to a temperature of 100°C for 1 minute using a temperature-regulated oven (Weiss-Gallenkamp). This temperature was selected as the heated pluggers used with the System B device reached to a maximum temperature of 100°C during obturation regardless of the setting on the liquid display (8, 9).

TABLE 1. Constituents of Sealers Supplied by the Manufacturers

	Component 1	Component 2
MTA Fillapex	MTA Base resin Silicon oxide Titanium dioxide	Salicylate resin Bismuth oxide Silicon oxide
Septodont Sealer	Tricalcium silicate Zirconium oxide	Water
Apexit Plus	Calcium hydroxide Hydrated collophonium Silicon oxide	Salicylate resin Bismuth oxide/ bismuth carbonate Silicon oxide
AH Plus	Bisphenol-A epoxy resin Bisphenol-F epoxy resin Iron oxide pigments Calcium tungstate Zirconium oxide Silicon oxide	Dibenzyldiamine Aminoadamantane Tricyclodecane-diamine Calcium tungstate Zirconium oxide Silicon oxide

Assessment of the Effect of Heat Application on Material Chemistry

The materials were mixed according to manufacturer's instructions and were compacted in cylindric rubber molds 15 mm in diameter and 2-mm high. Half the specimens were subjected to a temperature of 100°C for 1 minute, whereas the others were maintained at 37°C. All experiments were performed in triplicate.

X-ray Diffraction Analysis. Phase changes in the sealers subjected to different temperatures in the early stages of maturation were assessed. After subjecting the freshly mixed sealers to the different temperatures, they were placed in a climatic chamber (Weiss-Gallenkamp) and allowed to completely set at 37°C for 48 hours at 100% humidity. The sealers were then crushed to a very fine powder using an agate mortar and pestle. An X-ray diffractometer (Rigaku, Tokyo, Japan) with Cu Kα radiation at 40 mA and 45 kV was used, and the detector was set to rotate between 10° and 60°, with a sampling width of 0.05° and a scan speed of 1°/min at 15 rpm. Phase identification was accomplished with a search-match software using the International Centre for Diffraction Data database (International Centre for Diffraction Data, Newtown Square, PA).

Fourier Transform Infrared Spectroscopy. Fourier transform infrared (FT-IR) analysis of the sealers before and after the application of heat was performed by FT-IR spectroscopy (IRAffinity-1; Shimadzu Corp, Kyoto, Japan). To obtain the FT-IR spectra, the samples were powdered using a mortar and pestle, and 2–5 mg of each powder component was added to 100 mg potassium bromide and analyzed in the infrared spectrophotometer (Shimadzu IRAffinity) using transmitted infrared spectroscopy.

Assessment of the Effect of Heat Application on Physical Properties

The physical properties of the sealers before and after the application of heat were assessed. The setting time, flow, and film thickness were evaluated following ISO 6876 (10) specifications. All tests were performed in triplicate.

Assessment of Setting Time. The setting time of sealers was evaluated by dispensing the sealers into molds measuring 10 mm in diameter and 2-mm high. A stopwatch was started, and the molds were placed in an incubator at 37°C or initially kept at 100°C for 1 minute and then transferred to the incubator until the end of setting. Testing for setting was performed using a modified Vicat apparatus, which is similar to the Gilmore type suggested in ISO 6876, consisting of a weighted needle of square cross-section of side 2 $\pm$ 0.1 mm with a total mass of 100 $\pm$ 0.5 g. The square cross-section deviated from the cylindrical cross-section suggested by ISO 6876 (10). The sealers were considered to have set when the needle was lowered gently onto the material surface and did not leave a complete square indentation on it.

Flow. The materials were mixed and subjected to 37°C or 100°C for 1 minute to simulate the warm vertical compaction technique. Using a graduated pipette, 50 μL of the material were then dispensed on a glass plate measuring 40 $\times$ 40 mm and 5 mm in thickness. The second glass plate weighing 20 g was placed centrally on top of the sealer followed by the 100-g weight. The assembly was left in place for 10 minutes from the start of mixing, after which the maximum and minimum diameters of the compressed disc of sealer were measured using a micrometer. The mean diameter was calculated if the diameters agreed to within 1 mm. If not, the test was repeated.

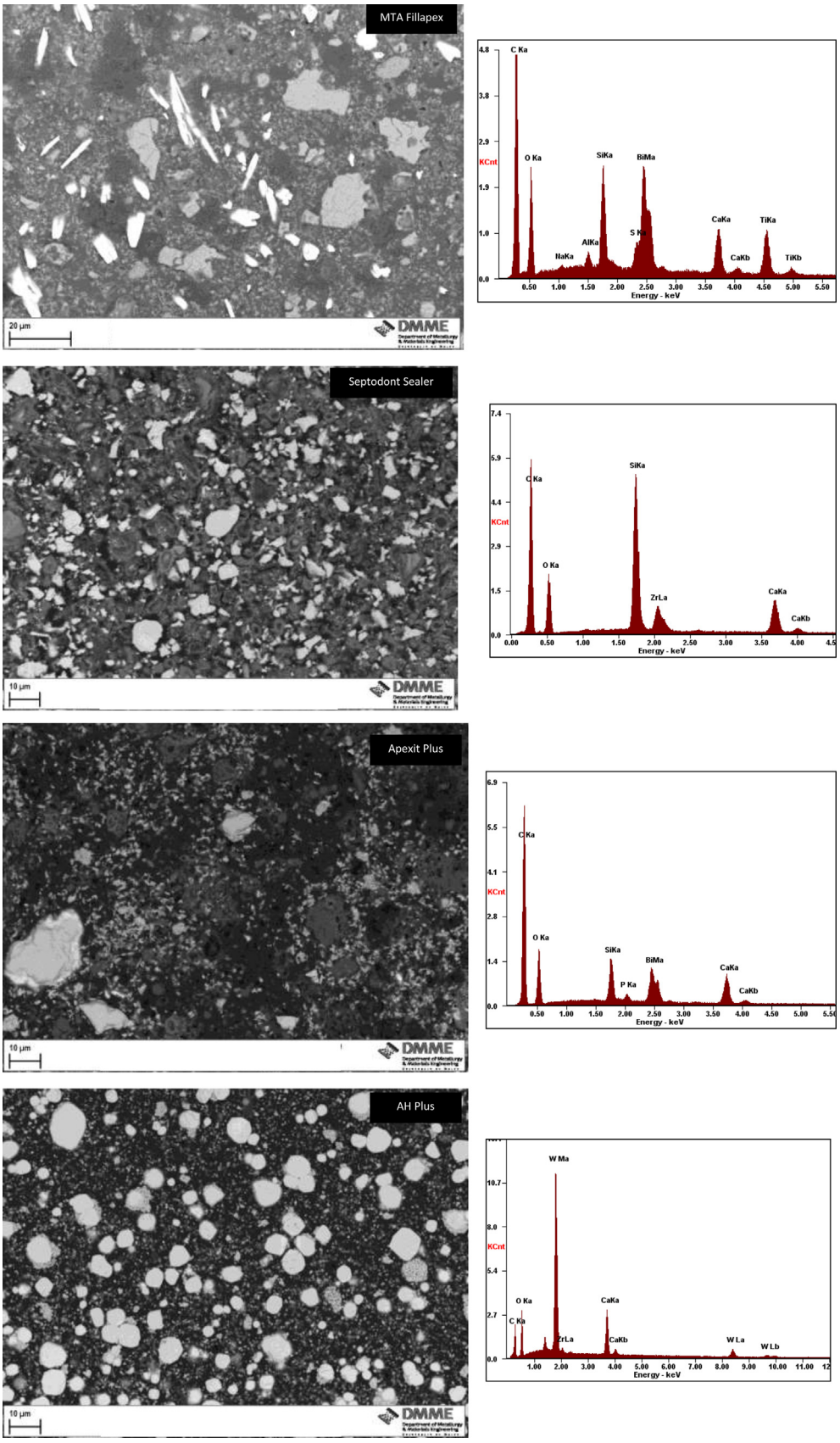

Figure 1. Backscatter scanning electron micrographs and energy-dispersive spectroscopic analysis determining material composition.

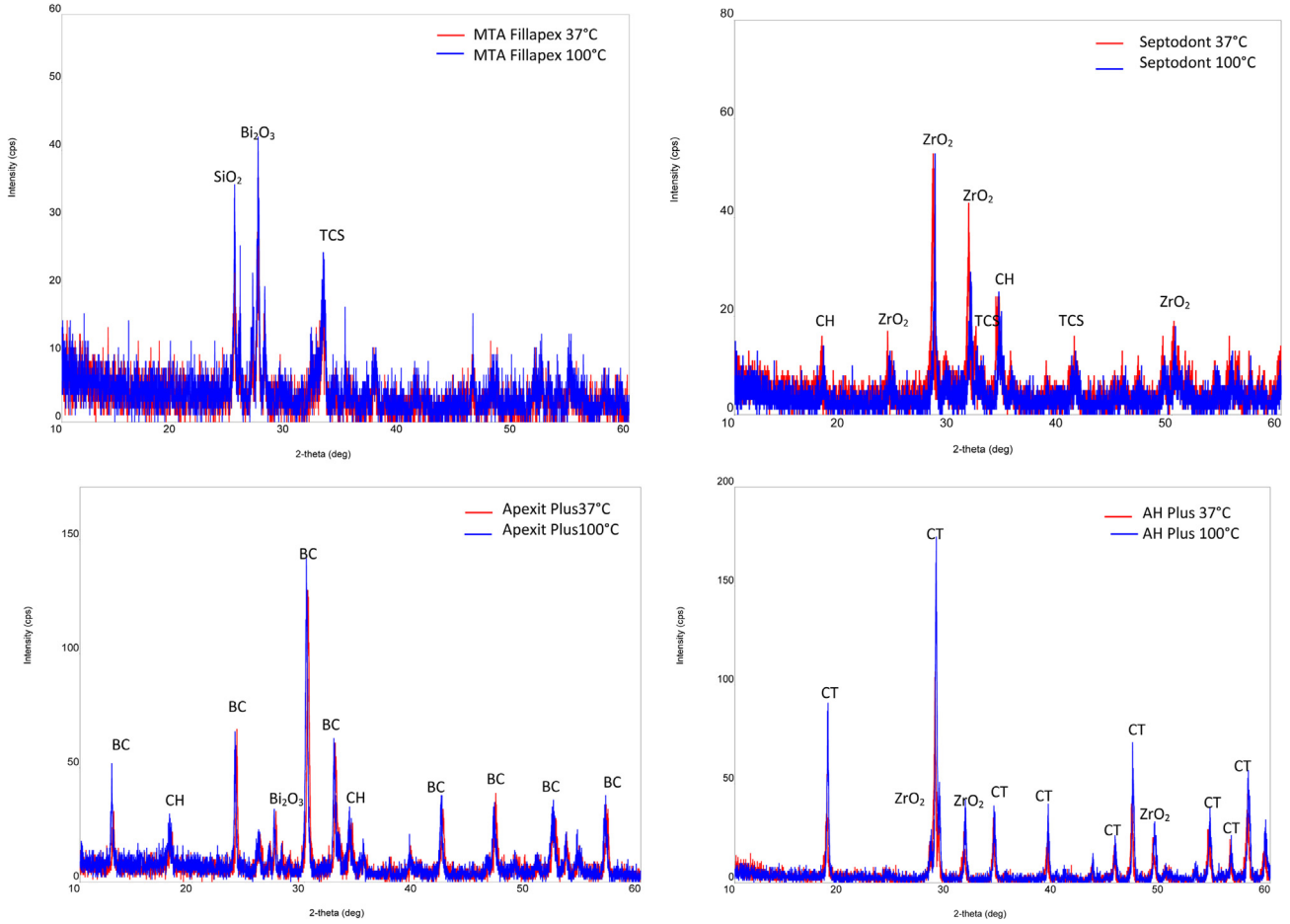

Figure 2. X-ray diffractograms of sealers subjected to 37°C and 100°C for 1 minute and incubated at 37°C and 100% humidity for 48 hours until fully set. BC, bismuth carbonate; CH, calcium hydroxide; CT, calcium tungstate; TCS, tricalcium silicate.

Film Thickness. For the film thickness test, the combined thickness of 2 glass plates each measuring 5 mm in thickness and having a surface area of 200 mm^2 was measured by using a micrometer to an accuracy of 1 μm. The materials were mixed, subjected to either 37°C or 100°C for 1 minute, and then placed on a glass plate and the other plate placed over the sealer and inserted in a loading device (Triaxial; ELE International, Leighton Buzzard, UK). A load of 150 N was applied until the sealer filled the area in between the glass plates. After 10 minutes from the start of mixing, the thickness of the combined glass plates and sealer was measured by using a micrometer.

Statistical Analysis

The data were evaluated using Statistical Package for the Social Sciences software (PASW Statistics 18; SPSS Inc, Chicago, IL). Parametric testing performed using the Kolmogorov-Smirnov test indicated that the data were normally distributed. Analysis of variance with $P = .05$ and the Tukey post hoc test were used to perform multiple comparison tests.

Results

Characterization of Materials by Scanning Electron Microscopy and EDS

The scanning electron micrographs and energy-dispersive spectroscopic analysis of the sealers are shown in Figure 1. The sealers exhibited a variety of particle sizes; they were lowest for Apexit and highest for MTA Fillapex, and both the cement particles and bismuth oxide showed large particle sizes. Energy-dispersive spectroscopic analysis of MTA Fillapex exhibited peaks for calcium, silicon, aluminum, sodium, and sulfur belonging to the cement particles, showing the Portland cement origin of the cementitious phase of the MTA Fillapex. Peaks of bismuth and titanium were also present. The cementitious phase of the novel sealer was composed of calcium, silicon, and oxygen whereas the shinier particles were composed of zirconium and oxygen, making up the radiopacifying material. Apexit Plus was composed of calcium, bismuth, phosphorus, silicon, and oxygen. AH Plus exhibited a resin matrix (rich in carbon) with 2 particles sizes. Energy-dispersive spectroscopic analysis exhibited peaks for calcium, zirconium, tungsten, and oxygen. The larger particles were composed of calcium and tungsten, whereas the smaller ones showed peaks for zirconium and oxygen.

Assessment of the Effect of Heat Application on Material Chemistry

Setting was analyzed by X-ray diffraction analysis of the sealers subjected to either 100°C or 37°C during the first minute of setting to simulate the heat changes sustained by the sealer during warm vertical compaction. The X-ray diffraction plots are shown in Figure 2. Tricalcium silicate hydrates by forming calcium silicate hydrate and calcium hydroxide. The calcium hydroxide peak is at 18°2θ when using a copper tube. Although MTA Fillapex is also based on tricalcium silicate, no calcium hydroxide was detected after the sealer set. On the other hand, a calcium hydroxide peak was evident in the experimental sealer. Apexit Plus also exhibited a calcium hydroxide peak. Bismuth oxide and

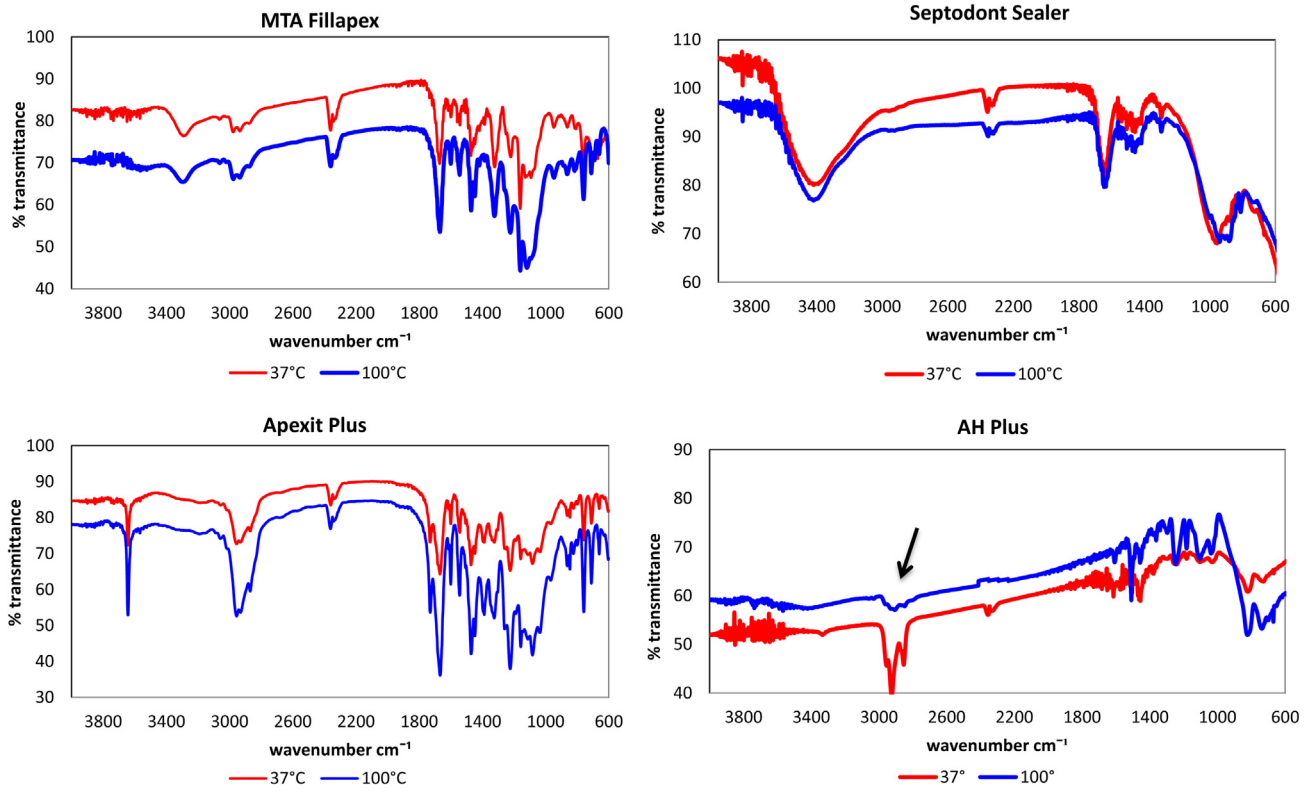

Figure 3. FT-IR spectroscopic plots of sealers subjected to 37°C and 100°C for 1 minute and incubated at 37°C and 100% humidity for 48 hours until fully set. The *arrow* shows the reduction in intensity of the stretching vibration of the N-H group present at 2900/cm.

bismuth carbonate peaks were also evident in Apexit Plus. The higher temperatures did not affect the crystalline phases of the sealers.

The infrared spectroscopy results are shown in Figure 3. The temperature did not change the MTA Fillapex, Septodont sealer, or Apexit Plus. Spectral changes were evident in the AH Plus sealer, which exhibited variations in the high-frequency part of the spectrum between 4000 and 1300/cm. The stretching vibration of the N-H group present at 2900/cm was reduced after heat was applied to AH Plus. AH Plus contains dibenzyldiamine, aminoadamantane, and tricyclodecane-diamine in paste B. The polyamines in paste B react with the epoxy resins in paste A, breaking the epoxide group bond and, as a consequence, forming polymers. Reduction in the polyamines caused by heat applied during warm vertical compaction will affect the polymerization process of AH Plus with resultant changes in the physical and mechanical properties of the resulting material.

Assessment of the Effect of Heat Application on Physical Properties

Table 2 shows the results for the setting time, flow, and film thickness for the 3 test sealers subjected to different temperatures during the first minute after the end of mixing. The experimental sealer had the shortest setting time (1.5 and 0.5 hours at 37°C and 5 minutes at 100°C) when compared with the other sealers ($P < .001$). AH Plus at 37°C exhibited the longest setting time (20.2 and 2.5 hours at 37°C and 5 minutes at 100°C). The high temperature did not change the setting time of MTA Fillapex (about 9 hours, $P = .999$) or the experimental sealer ($P = .255$). Heat early during the setting reaction reduced the setting time of AH Plus and Apexit Plus ($P = 0$). The Apexit Plus set after approximately 7 hours after the application of heat, whereas it required 37 hours at 37°C.

The ISO 6876 (10) standard for flow and film thickness requires a 17-mm flow diameter and a film thickness of less than 50 μm. AH Plus and the experimental sealer exhibited similar flow properties ($P = .67$), lower than that specified by ISO 6876 (10). MTA Fillapex and Apexit Plus met the ISO requirements. Temperature did not affect the flow characteristics of MTA Fillapex ($P = 1.00$), but a higher initial temperature reduced the flow of the experimental sealer ($P = 0$); an increased flow was exhibited with AH Plus, making it comply with the ISO specifications. Apexit Plus also exhibited an increase in flow after heating for 1 minute.

TABLE 2. Sealer Properties Tested in Accordance with ISO 6876 (2012) Specifications after Subjecting the Sealers to a Temperature of 37°C or 100°C for 1 Minute to Simulate Warm Vertical Compaction

	Setting time/hours		Flow/mm		Film thickness/μm	
Sealer	37°C	100°C	37°C	100°C	37°C	100°C
MTA Fillapex	9 ± 0.0	9 ± 0.1	25 ± 0.4	25 ± 1.1	33 ± 4.2	32 ± 0.7
Septodont Sealer	1.5 ± 0.1	0.5 ± 0.0	15 ± 0.3	6 ± 0.1	72 ± 11.3	349 ± 13.4
Apexit Plus	37 ± 3.5	7 ± 0.2	18 ± 0.4	19 ± 0.3	27 ± 11.3	27 ± 5.6
AH Plus	20 ± 1.3	2.5 ± 0.1	15 ± 0.3	18 ± 0.8	60 ± 9.1	175 ± 9.9

MTA Fillapex and Apexit Plus complied to ISO 6876 (10) with a film thickness less than 50 μm, whereas the experimental sealer and AH Plus did not meet the requirement. An initially higher temperature increased the film thickness of AH Plus (P = 0) and the experimental sealer (P = 0). MTA Fillapex and Apexit Plus were unaffected (P = 1.00).

Discussion

The materials investigated included 4 sealers. AH Plus, MTA Fillapex, and Apexit Plus are resin based, whereas AH Plus is epoxy resin based; the latter 2 sealers include a salicylate resin matrix as shown in Table 1. MTA Fillapex and the novel sealer were tricalcium silicate based containing different radiopacifiers (ie, bismuth oxide in MTA Fillapex and zirconium oxide in the novel sealer). Apexit Plus is calcium hydroxide based with both bismuth oxide and bismuth carbonate radiopacifiers. The calcium hydroxide in Apexit Plus is present in the unmixed material, whereas in the novel sealer it is formed as a byproduct of hydration reaction. The crystalline phases in each material were identified in the current study using X-ray diffraction analysis and elemental composition using scanning electron microscopic/energy-dispersive spectroscopic analysis.

Sealer properties after the application of heat for 1 minute were investigated. A temperature of 100°C was used to mimic the clinical situation. During warm vertical compaction, the heat is applied in continuous waves, and the full procedure usually lasts a minute. Although the heat carrier units are set to 200°C, the temperature in the heat carrier never rises to 200°C (7) and is approximately 100°C (8, 9). Compliance to ISO 6876 (10) was assessed after the application of heat.

The seal of the root canal obturation is improved when using the warm vertical compaction technique (11, 12). The properties of AH Plus sealer are modified when heated. AH Plus loses the amine groups with the application of heat. These amine groups are setting initiators and are necessary for the polymerization reaction to occur. The setting time of AH Plus was reduced considerably, and the film thickness increased. The heat accelerated the setting reaction, which led to an increase in the film thickness of the sealer. The same changes were observed in the novel sealer.

An increase in temperature accelerates the setting of tricalcium silicate–based materials, and this has been reported also for industrial Portland cement used in concrete (13). The material composition of the Septodont sealer was not modified by heat application as indicted by the X-ray diffraction and FT-IR analyses.

The changes to the material chemistry and physical properties of AH Plus when used during warm vertical compaction of gutta-percha have already been reported (8, 9). Although MTA Fillapex and Apexit Plus are also resin based, the heat generated during warm vertical compaction did not affect the material chemistry and physical characteristics. Thus, MTA Fillapex and Apexit Plus can be recommended as sealers for use with warm vertical compaction techniques. The setting time at body temperature reported for Apexit Plus (14, 15) is lower than the setting time in the current study.

Tricalcium silicate–based sealers have been developed because of the bioactivity shown by tricalcium silicate (16–18). The interaction with dentin fluid potentially induces biomineralization with the formation of mineral tags within the dentinal tubules, thus enhancing the biological activity within the root canal (19). The mineral infiltration zone has been reported with Biodentine (Septodont) in contact with dentin (20).

MTA Fillapex was among the first sealers manufactured based on mineral trioxide aggregate. Although several publications report the physical (21, 22), chemical, and biological (23–25) properties of MTA Fillapex, the information on the composition, hydration, and bioactivity is scarce. Although MTA Fillapex is a Portland cement–based material as shown by the scanning electron microscopic/energy-dispersive spectroscopic analysis, no formation of calcium hydroxide has been demonstrated. This is evident from the lack of the calcium hydroxide peak on X-ray diffraction analysis.

The experimental sealer exhibited the formation of calcium hydroxide early in the setting process. Thus, this sealer will probably promote bioactivity within the root canal and would be expected to interact with the dentin and form mineral tags. The formation of calcium hydroxide was not diminished by the heat applied in the early stages of setting.

Conclusions

The choice of sealer should be considered when selecting the obturation technique. The use of experimental tricalcium silicate–based sealer is recommended for obturations using cold laterally condensed gutta-percha. This novel sealer exhibits the formation of calcium hydroxide on hydration and thus would potentially promote bioactivity and adhesion to the canal wall through mineral tags. MTA Fillapex and Apexit Plus should be used with warm gutta-percha obturation techniques.

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

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