Characterization of heat resistant hydraulic sealer for warm vertical obturation

Hadis, Mohammed; Camilleri, Josette

DOI:

[10.1016/j.dental.2020.05.008](https://doi.org/10.1016/j.dental.2020.05.008)

License:

Creative Commons: Attribution-NonCommercial-NoDerivs (CC BY-NC-ND)

Document Version

Peer reviewed version

Citation for published version (Harvard):

Hadis, M & Camilleri, J 2020, 'Characterization of heat resistant hydraulic sealer for warm vertical obturation', *Dental Materials*, vol. 36, no. 9, pp. 1183-1189. <https://doi.org/10.1016/j.dental.2020.05.008>

[Link to publication on Research at Birmingham portal](#)

General rights

Unless a licence is specified above, all rights (including copyright and moral rights) in this document are retained by the authors and/or the copyright holders. The express permission of the copyright holder must be obtained for any use of this material other than for purposes permitted by law.

- Users may freely distribute the URL that is used to identify this publication.
- Users may download and/or print one copy of the publication from the University of Birmingham research portal for the purpose of private study or non-commercial research.
- User may use extracts from the document in line with the concept of 'fair dealing' under the Copyright, Designs and Patents Act 1988 (?)
- Users may not further distribute the material nor use it for the purposes of commercial gain.

Where a licence is displayed above, please note the terms and conditions of the licence govern your use of this document.

When citing, please reference the published version.

Take down policy

While the University of Birmingham exercises care and attention in making items available there are rare occasions when an item has been uploaded in error or has been deemed to be commercially or otherwise sensitive.

If you believe that this is the case for this document, please contact UBIRA@lists.bham.ac.uk providing details and we will remove access to the work immediately and investigate.

Characterization of heat resistant hydraulic sealer for warm vertical obturation

Mohammed Hadis, Josette Camilleri

*School of Dentistry, Institute of Clinical Sciences, College of Medical and Dental Sciences,
University of Birmingham, Birmingham, United Kingdom*

Key words: root canal sealers, warm vertical compaction, sealer characterisation

Correspondence

Josette Camilleri
School of Dentistry,
Institute of Clinical Sciences,
College of Medical and Dental Sciences,
University of Birmingham,
5, Mill Pool Way,
Edgbaston,
Birmingham, B5 7EG
United Kingdom
J.Camilleri@bham.ac.uk

Characterization of heat resistant hydraulic sealer for warm vertical obturation

Abstract

Introduction: Warm vertically compacted gutta-percha is a technique that is used by most specialist to obturate the root canal. The sealers currently available show deterioration when heated. This technique is popular as the heated gutta-percha is compacted vertically to take the shape of the root canal thus reducing any spaces and voids that will allow bacterial recontamination of the root canal space. New biologically active sealers that do not Most of the sealers used with gutta-percha in warm compaction obturation techniques sustain irreversible chemical and physical changes when heated are an attractive alternative to be used with warm vertical compaction obturation technique. Thus, new sealers that can be used with warm vertical compaction are being developed. The aim of this study was to measure the heat generated by warm vertical compactors inside the root canal, characterise a newly developed root canal sealer at different temperatures and verify its suitability at the actual temperature window used clinically, for use in the warm vertically compacted gutta-percha obturation technique.

Methodology: The typical temperatures generated by 2 heat carriers in a root canal were assessed by thermocouples. Two premixed root canal sealers TotalFill BC and HiFlow BC (FKG, Switzerland) were allowed to set and they were characterized by scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and X-ray diffraction analysis (XRD) immediately after setting and by XRD and after 28 days in physiological solution. The ion leaching in solution was assessed by inductively coupled plasma (ICP). The organic component was extracted in acetone and assessed by Fourier transform infrared spectroscopy (FT-IR) for both the unset sealer and sealer subjected to different

temperatures. ~~The typical temperatures generated by 2 heat carriers in a root canal were assessed by thermocouples.~~ The heat profiles of both sealers were at 50, 100 and 200°C was investigated by FT-IR ~~to evaluate the chemical changes to the sealers subjected to heat.~~ and thermographic analysis, was performed from 50-200°C on both sealers. ~~The organic component of both sealers after being subjected to no change in temperature and to an increase to 50, 100 and 200°C were characterized by FT-IR.~~

Results: None of the devices tested achieved the temperatures set on the dial. The highest temperatures were coronal followed by apical for both devices. The sealers were identical except for the vehicle. The inorganic components included tricalcium silicate, dicalcium silicate and zirconium oxide. No calcium hydroxide was produced by any of the sealers after immersion in physiological solution but calcium was released in solution. ~~None of the devices tested achieved the temperatures set on the dial. The highest temperatures were coronal followed by apical for both devices.~~ The chemistry of both sealers was modified when heated but both recovered when cooled. ~~The FT-IR profile of the two sealers was different when they were heated but both recovered when the heat was withdrawn.~~ ~~Extraction of the organic component showed that the heat did not affect both sealers. Both sealers exhibited a mass loss when heated which increased at higher temperatures.~~

Conclusions: The heat carriers were unreliable and the heat generated inside the canal was not the same as the temperature set on the dial. Since The highest temperature was generated coronally. ~~Both sealers had identical chemistry save for minimal modifications to the organic component and .Both sealers were resistant to heat and exhibited mass loss with increasing temperatures.~~ TotalFill BC sealer is ~~thus~~ recommended for use with warm vertical compaction technique as it is cheaper and just as effective. ~~There is some concern~~

~~with the sealer setting as no calcium hydroxide was formed and this limits the chemical effectiveness.~~

1. Introduction

The warm vertical compaction of gutta-percha is a very popular obturation technique where the gutta-percha is heated until it changes phase and softens, then it is compacted vertically to take the shape of the prepared root canal [1]. With this technique the obturation is composed mostly of gutta-percha with minimal amounts of sealer. There is no specific sealer that is suggested for use with this technique so all sealer types are thought to be suitable.

The temperature of phase changes to gutta-percha have been reported to be 60°C to convert from the β form to completely amorphous [21, 32]. Regardless of this, all the heat carriers indicate the use of temperature settings ranging from 180-230°C. There seems to be no scientific reason for the use of such temperatures which are unsafe. Increases in temperature of bone has been shown and also will lead to lead to bone necrosis as the bone can only tolerate an increase by few degree [4]. Luckily enough Regardless of the reading on the dial, the temperature of the heat carriers is less [35, 46] and the temperature

is further dissipated when the heat carrier is placed in a tooth [46, 57] and sealer is used in the root canal [46]. As a result of this, tThe temperature rise on the external surface of the root is minimal [35, 46, 68].

A rise in temperature in the root canal has deleterious effects on the root canal sealers particularly the epoxy resin-based sealers such as AH Plus (Dentsply). At higher temperatures the AH Plus showed irreversible changes in chemistry [35, 46, 79, 810] and its physical properties were modified with a reduction in the setting time and flow and increase in film thickness [35, 46, 810-129]. Zinc oxide eugenol-based sealers sustained an increase in setting time when exposed to heat but the flow was unaffected [911] while BioRoot RCS showed changes in its chemistry due to the water evaporation at high temperature [8, 10] and iRoot SP had reduced flow [911]. Heating of AH Plus also resulted in an increase in void volume while BioRoot RCS and Gutta-flow were unaffected [129].

Recently a new hydraulic calcium silicate-based sealer which can be used with warm vertical compaction has been developed. This sealer is claimed to have a similar chemistry to the premixed hydraulic sealers (BC sealers) manufactured by Brasseler/FKG Dentaire but is resistant to heat thus is recommended for use with warm vertical compaction obturation technique. ~~The sealer (HiFlow) is more expensive than the classical formulation of the BC sealer as although sold for the same price the syringe is smaller.~~ The aim of this research was to determine the temperature generated by two heat carriers inside the root canal in a simulated clinical environment and to characterise both the classical BC sealer and the HiFlow and assess their heat profiles and chemical changes when subjected to high

temperatures. ~~To date, both sealers have not been investigated and characterised when subjected to heat.~~

2. Materials and methods

In this study two premixed hydraulic calcium silicate sealers are tested. These include TotalFill and HiFlow BC sealers. Both sealers are manufactured by FKG Dentaire (Chaux-de-Fonds, Switzerland). The HiFlow was developed as an alternative to the BC sealer to be used together with gutta-percha for warm vertical compaction. ~~All analysis was undertaken in triplicate.~~

2.12 Assessment of heat profiles of the endodontic heat carriers

The heat profiles of two heat carriers was assessed using a method described in the literature [57]. A single rooted tooth was accessed and chemo-mechanically prepared with ProTaper Gold to a size F2 using sodium hypochlorite irrigation. Three holes 0.8 mm in diameter were drilled along the root face 2 mm, 8 mm and 12 mm from the apex. K-type thermocouples (0.3 mm diameter, Maplins, UK) were inserted into the root canal through these holes and were held in place by unfilled resin. The thermocouples were connected to a multi-channel data logger (PicoData Logger, TC-08, UK). The whole assembly was placed in alginate to simulate the clinical environment. Temperature was recorded in real-time at a rate of 1s⁻¹ for 30s using the PicoData logging software. The heat profiles generated by each device were composed of a 10 s pre-heating phase, 4-second heating phase followed by a

10-second cooling phase. Two endodontic heat carriers were tested; E&Q Master (Meta Biomed, Chalfont, PA, USA) and System-B (Orange, CA, USA) both using a fine tip inserted 3 mm short of the apex. The E&Q Master was operated at 180°C and 230°C with automatic cut-out after 4 seconds. System-B was set at 200°C and a heating cycle of 4 s heating and 10s cooling was recorded. Each cycle was repeated three times for each heat carrier and set temperature to check for repeatability and the data expressed as mean and standard deviation to the mean.

2.1-2 Sealer characterization

The organic component of the sealers was extracted in acetone by centrifugation from the unset sealer and analysed by Fourier transform infrared (FT-IR) spectroscopy. The set sealers were characterized by scanning electron microscopy, and X-ray diffraction (XRD) analysis immediately after setting and by XRD after 28 days immersed in Hank's balanced salt solution (HBSS) at 37°C. The leachate was tested by inductively coupled plasma mass spectroscopy (ICP-MS). All experiments were carried out in triplicate

The sealers were syringed and specimens 10 mm in diameter and 2 mm thick were made.

The specimens were covered by a wet gauze and were placed in an incubator at 37°C and 100% humidity for 24 hours. Setting was verified by indenting the specimen surfaces. One group were tested immediately after setting and the rest were placed in 5 mL of HBSS for 28 days.

For scanning electron microscopy, the specimens were embedded in a cold cure epoxy resin (Epoxy-fix, Struers, Ballerup, Denmark) and the surfaces were polished with an automatic polishing machine (Buhler, Lake Buff, USA) using 250, 500 and 1200 grit diamond discs under water coolant (Struers, Ballerup, Denmark) and polishing cloths (Struers) with ascending grades of diamond-impregnated polishing pastes (Struers). Once polished the embedded specimens were mounted on aluminium stubs and held in place with carbon tape and were sputter coated with gold. The specimens were then viewed under the SEM (Zeiss). The sealers were assessed in back scatter mode to obtain elemental contrast. Images at different magnifications were obtained. Energy dispersive spectroscopy was performed to assess the elemental distribution.

For the XRD the aged specimens were retrieved from solution and dried in desiccator. For both ages, the sealers were then ground using a mortar and pestle to a fine powder. XRD was performed with a diffractometer (Bruker D8 Advance, Bruker Corp., Billerica, MA, USA) with a CuK α radiation at 40 mA and 45 kV was set to rotate between 10-60° with a 0.02° 2 θ step and a step time of 0.6 s. Phase identification was undertaken using a search-match software using the ICDD database (International Centre for Diffraction Data, Newtown Square, PA, USA). The leachates were assessed for calcium, silicon, zirconium and phosphorus with inductively coupled plasma-optical emission spectrometry (Optima 8000, Perkin Elmer, Waltham, USA). Calculation of results was performed in parts per million (ppm).

~~2.2 Assessment of heat profiles of the endodontic heat carriers~~

The heat profiles of two heat carriers was assessed using a method described in the literature [5]. A single rooted tooth was accessed and chemo-mechanically prepared with ProTaper Gold to a size F2 using sodium hypochlorite irrigation. Three holes 0.8 mm in diameter were drilled along the root face 2 mm, 8 mm and 12 mm from the apex. K-type thermocouples (0.3 mm diameter, Maplins, UK) were inserted into the root canal through these holes and were held in place by unfilled resin. The thermocouples were connected to a multi-channel data logger (PicoData Logger, TC-08, UK). The whole assembly was placed in alginate to simulate the clinical environment. Temperature was recorded in real time at a rate of 1s^{-1} for 30s using the PicoData logging software. The heat profiles generated by each device were composed of a 10 s pre-heating phase, 4 second heating phase followed by a 10 second cooling phase. Two endodontic heat carriers were tested; E&Q Master (Meta Biomed, Chalfont, PA, USA) and System-B (Orange, CA, USA) both using a fine tip inserted 3 mm short of the apex. The E&Q Master was operated at 180°C and 220°C with automatic cut-out after 4 seconds. System-B was set at 200°C and a heating cycle of 4 s heating and 10s cooling was recorded.

2.3 Investigation of the chemical changes in the sealers

The sealers were dispensed on a heated diamond plate (MIRacle) attached to a control module (Pike Technologies, USA). The chemical changes to the sealers exposed to a heating and cooling cycle were assessed by FT-IR in ATR mode as described for the sealer characterization. A baseline measurement was taken immediately after sample placement at room temperature ($21 \pm 1^\circ\text{C}$) and then the heating module was activated to heat the samples at a rate of $12.5^\circ\text{C}/\text{min}$. Measurements were taken from 25°C to either 50, 100 or

200°C at increments of 25°C and then air-cooled to 25°C and re-measured. All spectra were baseline corrected between 650-4000 cm⁻¹ and an average (n = 3) was taken. The testing assembly was kept at high relative humidity.

Changes in weight sustained at different temperatures were recorded by thermogravimetric analysis/differential scanning calorimetry, (TGA/DSC; Mettler-Toledo, Beaumont Leys, Leicester, UK). 20 mg of set sealers were tested at a temperature range of 50°C - 200°C at a heating rate of 10°C min⁻¹ and O₂ flow rate of 40 ml min⁻¹. The experiment was repeated three times to ensure repeatability.

The organic component was also extracted after setting at room temperature and after subjecting the sealers to temperatures of 50, 100 and 200 °C and this was tested by FT-IR (n = 3).

2.4 Statistical analysis

The statistical analysis was performed using Predictive Analytics Software (PASW) version 18. One-way ANOVA was used to determine whether there were significant differences between data sets. The data was tested to ensure it was normally distributed and then with analysis of variance with P = 0.05 the turkey post hoc test was used.

3. Results

3.12 Assessment of heat profiles of endodontic heat carriers

The heat generated by the endodontic heat carriers is shown in Table 12. The higher setting of the EVO carrier led to a higher temperature mostly coronally with minimal

Formatted: Font: Italic

Formatted: Normal, No bullets or numbering

Formatted: Font: Italic

changes mid-root and apically. None of the heating devices achieved the temperature set on the dial. All devices exhibited the highest temperature at the coronal portion followed by the apical region with the mid-root exhibiting the lowest temperature. The highest temperature was recorded coronally for the EVO set at 230°C.

~~3.2.1~~ *Sealer characterization*

The FT-IR scans showing the extraction of the organic component for both sealers is shown in Figure 1a. The organic component for the sealers was different as it had a different FT-IR signature. The scanning electron micrographs and EDS plots for or both sealers are shown in Figure 1b. Both sealers had a similar morphology consisting of a composite microstructure with particles of various sizes and opacity. Both materials were composed of calcium, silicon, zirconium and oxygen.

The XRD scans immediately after setting and after 28-day immersion in HBSS are shown in Figure 1c, and leachate analysis in Table ~~2.1~~. These were identical for both material types. The XRD plots showed predominant peaks of zirconia (ICDD 00-065-0687), tricalcium and dicalcium silicate (ICDD 00-001-1024, 01-070-2450). The leachate analysis (Table ~~1.2~~) showed calcium ion release for both materials with depletion in phosphorus amounts which can account for the taking up of the phosphorus by the materials from the HBSS. The zirconium was below detection limits and low levels of silicon were released.

~~3.2~~ *Assessment of heat profiles of endodontic heat carriers*

~~The heat generated by the endodontic heat carriers is shown in Table 2. The higher setting of the EVO carrier led to a higher temperature mostly coronally with minimal changes mid-~~

root and apically. None of the heating devices achieved the temperature set on the dial. All devices exhibited the highest temperature at the coronal portion followed by the apical region with the mid-root exhibiting the lowest temperature. The highest temperature was recorded coronally for the EVO set at 230°C.

3.3 Investigation of the chemical changes in the sealers

The thermal profiles of the materials while heating to either 50, 100 or 200°C and cooling are shown in Figure 2a. The sealers had a different FT-IR signature indicating a difference in chemistry even at room temperature. Both sealers sustained changes in the region of 2200 cm^{-1} which were enhanced the higher the temperature. For both sealers these and the changes at 2000 cm^{-1} for the TotalFill BC and at 1800 cm^{-1} for the HiFlow were reversible.

The TGA data is shown in Figure 2b. Both sealers showed a mass loss when heated. The mass loss increased with increasing temperatures. Extraction of organic component from set sealers after mixing at room temperature and after being subjected to high temperatures is shown in Figure 2c. The peaks correspond mainly to the acetone so not much of the vehicle could be extracted after setting and heating.

4. Discussion

The current study investigates 2-two sealer types manufactured by the same company. The TotalFill BC sealer has been characterised previously [134]. HiFlow BC sealer has been launched recently and there is very limited information available for this sealer. All the information regarding its composition is available on the company safety data sheet [14] and the safety data sheet provided is the same for both sealers. The manufacturer

recommends the HiFlow for use with warm vertical compaction obturation technique while the Totalfill BC sealer for single cone obturation technique [15]. The effectiveness of the single cone obturation technique with premixed hydraulic sealers has been verified clinically [16] and a recent survey of clinicians has also endorsed the popularity of this technique [17].

Since HiFlow had not been previously characterized, a full characterization using standard methods such as scanning electron microscopy, energy dispersive spectroscopy and X-ray diffraction analysis was undertaken in conjunction with leachate analysis and extraction of the organic component and analysis. This enabled the identification of the inorganic phases which were shown to be similar to those of Totalfill BC sealer as both sealers were composed mainly of tricalcium silicate and zirconium oxide. [13]. The chemistry of the inorganic component was not listed on the safety data sheet [14]. The extraction and analysis of the inorganic components showed a clear difference in the inorganic component chemistry between the sealers. The sealer chemical similarity has been confirmed in a recent publication [18] which included their identical biological behaviour. The only difference was the flow and radiopacity [11, 18]. currently there is no published data regarding this sealer type. The composition of TotalFill BC sealer is in accordance to previous literature [11] as the sealer was composed mainly of tricalcium silicate and zirconium oxide. The HiFlow BC sealer had an identical composition and both sealers had the same hydration profile.

The analysis undertaken showed the material composition rather than the surface chemistry in contact with physiological solution. Apatite nucleation over the materials was not assessed as this phenomenon was shown to be present only *in vitro* and cannot be

Formatted: Font: Italic

translated clinically [19]. All *ex vivo* analysis has shown the formation of calcium carbonate when hydraulic cements interacted with a clinical environment [20] and even in animal models [21]. A plausible scientific explanation has been reported [22] and the formation of calcium carbonate *in vivo* has also been recognized for bioglass which is also a bioactive material [23].

Formatted: Font: Italic

Formatted: Font: Italic

Formatted: Font: Italic

In the current research, nNo calcium hydroxide was observed on setting but some calcium ions were released in solution. The lack of formation of calcium hydroxide indicates the limited-slower hydration of this sealer in the early ages which is due to the time required for the organic component to be replaced with reaction by product form the hydration of the tricalcium silicate with water taken up from the surroundings. The limited hydration can be problematic as the calcium ion release by hydraulic cements has been linked to the antimicrobial properties-[24]of hydraulic sealers depends on the formation of the calcium hydroxide. The use of antimicrobial agents for irrigation has been shown to help with reduction in the microbial load on root canal obturating materials [25]. The biological assessment of both sealers also indicated an initial decreased cell viability which can be correlated to the release of calcium hydroxide which is lower than that of other similar sealer types [13] indicating the limitation of the alternative vehicle in allowing the release of calcium and its crystallization when compared to water-based hydraulic sealers [13].The only difference in the sealers seems to be the organic vehicle used. The literature provided with the materials is identical and the composition of the organic component is not divulged.

The heat carriers exhibited a different heat output to the setting on the dial. This has been reported previously [5, 6, 8, 26] and is a cause of concern since the temperature cannot be controlled. In accordance with previous research, ~~t~~The highest temperatures were generated at the coronal aspect of the root canal [5] and ~~-This was in accordance to previous research [3].~~ ~~T~~The temperatures of the heat carriers recorded inside the root canal ranged from 45-70°C are similar to previous investigations [57]. ~~however in the current study the temperatures were not recorded in real time while obturating as the sealer would inadvertently block the reading of the thermocouple leading to erroneous results.~~ Furthermore, in the current study the sealers were also characterised to investigate the effect of the temperature changes being generated inside the root canal unlike the previous study where no sealer characterisation was carried out [5], thus in that study there was no way of knowing if the sealer chemistry was affected by the heat generated.

Based on the temperatures recorded inside the root canal, both sealers were subjected to heat starting at 50°C which was the temperature recorded in the apical portion where most of the sealer is located during warm vertical compaction of gutta-percha. The evaluations at 100°C were undertaken to be able to compare to previous studies on sealers ~~[35, 46, 79-~~ 1012] and the 200°C was assessed since the heat carriers were set at this temperature. The temperature applied in the current study was transient to avoid lengthy heat applications which are not clinically valid and also because the frequency and time of heat application effect the sealer chemistry [9]. ~~Although the HIFlow has been marketed as a newer generation to the TotalFill BC sealer, the latter had not been investigated when subjected to high temperatures except for showing a change in flow when heated [9].~~

Both sealers sustained some changes when exposed to heat that intensified with higher temperatures but all changes were reversible indicating that both sealers can be heated even to 200°C. ~~The Totalfill BC sealer had already been assessed at high temperatures and it showed a similar heat resistance to the current study [26]. The mass loss with increased temperature was minimal and would be stabilized in the long term by the fluid imbibition in the root canal as the hydraulic sealers interact with the dentine and dentinal fluids in the long term. The unavailability of the organic component to extraction when the sealer had set indicates the setting and the replacement of the organic component with reaction by-product as a result of the hydration initiated by the surrounding liquids. The heat generated during the warm vertical compaction did not affect the chemistry of the set sealers not even the organic component.~~

Conclusions

The heat carriers were unreliable and the heat generated inside the canal was not the same as the temperature set on the dial. ~~The highest temperature was generated coronally. Both sealers were resistant to heat and had identical chemistry-chemistries save except for minimal~~ modifications to the organic component. ~~Both sealers were resistant to heat.~~ TotalFill BC sealer is ~~thus~~ recommended for use with warm vertical compaction technique as it is cheaper and just as effective. ~~There is some concern with the sealer setting as no calcium hydroxide was formed and this limits the chemical effectiveness.~~

Acknowledgements

Dr Jiangio Liu for his assistance with the testing and Prof Brian Darvell for his guidance with the chemical analysis.

Declaration

The authors ~~deny~~ declare no ~~any~~ conflict of interest.

References

1. Schilder H. Filling root canals in three dimensions. Dent Clin North Am. 1967

Nov;723-44.

2. Marlin J, Schilder H. Physical properties of gutta-percha when subjected to heat and vertical condensation. Oral Surg Oral Med Oral Pathol. 1973

Dec;36(6):872-9.

3. Schilder H, Goodman A, Aldrich W. The thermomechanical properties of gutta-percha. 3. Determination of phase transition temperatures for gutta-percha. Oral Surg Oral Med Oral Pathol. 1974 Jul;38(1):109-14.

4. Eriksson AR, Albrektsson T. Temperature threshold levels for heat-induced bone tissue injury: a vital-microscopic study in the rabbit. J Prosthet Dent 1983;50:

101-7.

5. Viapiana R, Baluci CA, Tanomaru-Filho M, Camilleri J. Investigation of chemical changes in sealers during application of the warm vertical compaction technique. Int Endod J. 2015 Jan;48(1):16-27.

Formatted: Normal, No bullets or numbering

- ~~4-6.~~ Viapiana R, Guerreiro-Tanomaru JM, Tanomaru-Filho M, Camilleri J. Investigation of the effect of sealer use on the heat generated at the external root surface during root canal obturation using warm vertical compaction technique with System B heat source. J Endod. 2014 Apr;40(4):555-61.
- ~~5-7.~~ Donnermeyer D, Schäfer E, Bürklein S. Real-time Intracanal Temperature Measurement During Different Obturation Techniques. J Endod. 2018 Dec;44(12):1832-1836.
- ~~6-8.~~ Venturi M, Pasquantonio G, Falconi M, Breschi L. Temperature change within gutta-percha induced by the System-B Heat Source. Int Endod J. 2002 Sep;35(9):740-6.
- ~~7-9.~~ Atmeh AR, AlShwaimi E. The Effect of Heating Time and Temperature on Epoxy Resin and Calcium Silicate-based Endodontic Sealers. J Endod. 2017 Dec;43(12):2112-2118.
- ~~8-10.~~ Camilleri J. Sealers and warm gutta-percha obturation techniques. J Endod. 2015 Jan;41(1):72-8.
- ~~11.~~ Qu W, Bai W, Liang YH, Gao XJ. Influence of Warm Vertical Compaction Technique on Physical Properties of Root Canal Sealers. J Endod. 2016 Dec;42(12):1829-1833.
- ~~9.~~ Heran J, Khalid S, Albaaj F, Tomson PL, Camilleri J. The single cone obturation technique with a modified warm filler. J Dent. 2019 Oct;89:103181.
- ~~12.~~
- ~~13.~~ Xuereb M, Vella P, Damidot D, Sammut CV, Camilleri J. In situ assessment of the setting of tricalcium silicate-based sealers using a dentin pressure model. J Endod. 2015 Jan;41(1):111-24.

14. https://www.fkg.ch/sites/default/files/FKG_TotalFill%20BC%20Sealer_Safety%20data%20sheet.pdf

Formatted: Font: (Default) +Body (Calibri)

Formatted: Font: Font color: Black

15. <https://brasselerusadental.com/products/endosequence-bc-sealer-hiflow/>

16. Chybowski EA, Glickman GN, Patel Y, Fleury A, Solomon E, He J. Clinical Outcome of Non-Surgical Root Canal Treatment Using a Single-cone Technique with Endosequence Bioceramic Sealer: A Retrospective Analysis. J Endod. 2018 Jun;44(6):941-945.

17. Guivarc'h M, Jeanneau C, Giraud T, Pommel L, About I, Azim AA, Bukiet F. An international survey on the use of calcium silicate-based sealers in non-surgical endodontic treatment. Clin Oral Investig. 2020 Jan;24(1):417-424.

~~10-18.~~ Chen B, Haapasalo M, Mobuchon C, Li X, Ma J, Shen Y. Cytotoxicity and the Effect of Temperature on Physical Properties and Chemical Composition of a New Calcium Silicate-based Root Canal Sealer. J Endod. 2020 Apr;46(4):531-538.

19. Meschi N, Li X, Van Gorp G, Camilleri J, Van Meerbeek B, Lambrechts P. Bioactivity potential of Portland cement in regenerative endodontic procedures: From clinic to lab. Dent Mater. 2019 Sep;35(9):1342-1350.

20. Moizadeh AT, Aznar Portoles C, Schembri Wismayer P, Camilleri J. Bioactivity Potential of EndoSequence BC RRM Putty. J Endod. 2016 Apr;42(4):615-21.

21. Schembri Wismayer P, Lung CY, Rappa F, Cappello F, Camilleri J. Assessment of the interaction of Portland cement-based materials with blood and tissue fluids using an animal model. Sci Rep. 2016 Sep 29;6:34547. doi: 10.1038/srep34547.

22. Bohner M, Lemaître J. Can bioactivity be tested in vitro with SBF solution? Biomaterials. 2009;30:2175-9.

23. Mozafari M, Banijamali S, Bairo F, Kargozar S, Hill RG. Calcium carbonate: Adored and ignored in bioactivity assessment. *Acta Biomater.* 2019 Jun;91:35-47.
24. Koutroulis A, Kuehne SA, Cooper PR, Camilleri J. The role of calcium ion release on biocompatibility and antimicrobial properties of hydraulic cements. *Sci Rep.* 2019 Dec 13;9(1):19019. doi: 10.1038/s41598-019-55288-3.
25. Xu J, He J, Shen Y, Zhou X, Huang D, Gao Y, Haapasalo M. Influence of Endodontic Procedure on the Adherence of *Enterococcus faecalis*. *J Endod.* 2019 Jul;45(7):943-949.
26. Atmeh AR, Hadis M, Camilleri J. Real-time chemical analysis of root filling materials with heating: guidelines for safe temperature levels. *Int Endod J.* 2020 Jan 18. doi: 10.1111/iej.13269. [Epub ahead of print] Heran J, Khalid S, Albaaj F, Tomson PL, Camilleri J. The single cone obturation technique with a modified warm filler. *J Dent.* 2019 Oct;89:103181.
- 11.1.
12. Xuereb M, Vella P, Damidot D, Sammut CV, Camilleri J. In situ assessment of the setting of tricalcium silicate-based sealers using a dentin pressure model. *J Endod.* 2015 Jan;41(1):111-24.

Table 1: Leachate analysis for both sealers showing the concentration of ions leached in solution after 28-day exposure to Hank's balanced salt solution

Sealer	Ion concentration ppm			
	Ca	Zr	Si	P
BC-Sealer	867.87	BDL	2.64	-0.22
HiFlow	876.95	BDL	BDL	-0.39

BDL: below detection limits

Table 12: Temperatures recorded at different levels in the root canal with the various devices and settings

Formatted: Font: (Default) +Body (Calibri)

Formatted: Line spacing: Double, Numbered + Level: 1 + Numbering Style: 1, 2, 3, ... + Start at: 1 + Alignment: Left + Aligned at: 0.3" + Indent at: 0.55"

Formatted: Font: Font color: Black

Formatted: Indent: Left: 0.5", Line spacing: single, No bullets or numbering

Formatted: List Paragraph, Line spacing: single

Formatted: Centered

Device	Temperature °C		
	Coronal	Mid-root	Apical
EVO 180	65.3 ± 0.7	43.1 ± 0.2	46.2 ± 0.3
EVO 230	71.0 ± 0.4	45.02 ± 0.3	49.1 ± 0.4
System B	59.4 ± 0.9	42.2 ± 0.5	45.4 ± 0.7

Table 21: Leachate analysis for both sealers showing the concentration of ions leached in solution after 28-day exposure to Hank's balanced salt solution

<u>Sealer</u>	<u>Ion concentration ppm</u>			
	<u>Ca</u>	<u>Zr</u>	<u>Si</u>	<u>P</u>
<u>BC Sealer</u>	<u>867.87</u>	<u>BDL</u>	<u>2.64</u>	<u>-0.22</u>
<u>HiFlow</u>	<u>876.95</u>	<u>BDL</u>	<u>BDL</u>	<u>-0.39</u>

BDL: below detection limits

Figure

Figure 1a: FT-IR scan of the organic component extracted from both sealers using acetone and centrifugation

Figure 1b: Back scatter scanning electron micrographs and EDS scans of (a) TotalFill BC sealer and (b) HiFlow BC sealer showing microstructure and elemental composition

Figure 1c: XRD plots of both sealers immediately after setting and after 28 days immersed in HBSS showing the similar phases present in the materials [DCS: dicalcium silicate; TCS: tricalcium silicate; Z: zirconium oxide]

Figure

Figure 2a: FT-IR plots of both sealers exposed to different temperatures showing the chemical changes sustained during heating and cooling.

Figure 2b: Mass loss (black line) and heat flow (red line) of both sealers calculated by thermographic analysis

Figure 2c: FT-IR plots of the organic component of both sealers extracted in acetone after the sealer set at room temperature and after being subjected to different temperatures