BASIC RESEARCH – TECHNOLOGY

Evaluation of Physicochemical Properties of a New Calcium Silicate–based Sealer, Bio-C Sealer

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ABSTRACT

Introduction: Calcium silicate–based materials have been proposed as root canal sealers for root canal treatment. The aim of this study was to evaluate the physicochemical properties of a new calcium silicate–based sealer (Bio-C Sealer; Angelus, PR, Brazil) compared with a calcium silicate endodontic sealer (TotalFill BC Sealer; FKG Dentaire SA, La Chaux-de-Fonds, Switzerland) and an epoxy resin sealer (AH Plus; Dentsply DeTrey, Konstanz, Germany). **Methods:** The setting time and flow were evaluated based on ISO 6876 standard. The pH value was evaluated after different time intervals of storage in deionized water (1, 7, 14, and 21 days). Radiopacity was evaluated by radiographic analysis in millimeters of aluminum. Solubility and volumetric change were evaluated after 30 days of immersion in distilled water. Solubility was assessed by mass loss (%), and volumetric change was evaluated by micro–computed tomographic imaging. The data were submitted to analysis of variance and Tukey statistical tests ($P < .05$). **Results:** TotalFill BC Sealer and Bio-C Sealer were similar regarding radiopacity, volumetric change, and pH values ($P > .05$). Bio-C Sealer presented the shortest setting time and the highest flow and solubility ($P < .05$). AH Plus showed the highest radiopacity and the lowest flow, pH, solubility, and volumetric change ($P < .05$). **Conclusions:** Bio-C Sealer showed a short setting time, alkalinization ability, and adequate flow and radiopacity as well as low volumetric change. However, this sealer had higher solubility than the rates required by ISO 6876 standard. *(J Endod 2019;■:1–5.)*

KEY WORDS

Calcium silicate; endodontics; micro–computed tomography; physicochemical properties; root canal sealer

SIGNIFICANCE

Bio-C Sealer is a new calcium silicate–based root canal sealer that has an adequate setting time, flow, and radiopacity. Although this material had high solubility, the micro-CT assessment indicated low volumetric change.

Root canal filling is important for the long-term success of endodontic treatment[1]. Therefore, root canal sealers should have the appropriate physical and chemical properties[1] to achieve tridimensional sealing[2].

AH Plus (Dentsply DeTrey, Konstanz, Germany) is an epoxy resin–based root canal sealer considered the gold standard for its physicochemical properties[3]. However, the main limitation of AH Plus is its absence of bioactive properties[4]. Bioactive materials favor the periapical healing process[4]. Therefore, new calcium silicate–based endodontic sealers have been developed based on their excellent biological properties[5] and bioactive potential[4]. Premixed ready-to-use calcium silicate–based sealers present biocompatibility and bioactivity, and their radiopacity and flow comply with ISO 6876:2012. These sealers promote high pH, allow Ca^{2+} ion release, and present bond strength similar to AH Plus[3]. However, high solubility is also reported for ready-to-use calcium silicate–based endodontic sealers[3]. Thus, studies evaluating the physicochemical properties of newly developed calcium silicate–based materials are needed before considering their clinical applications[6].

TotalFill BC Sealer (FKG Dentaire SA, La Chaux-de-Fonds, Switzerland) is a calcium silicate–based material that presents adequate physicochemical[7,8] and biological properties[8,9] as well as an antimicrobial effect[9]. Bio-C Sealer (Angelus, Londrina, PR, Brazil) is a new root canal sealer containing calcium silicates, calcium aluminate, calcium oxide, zirconium oxide, iron oxide, silicon dioxide, and dispersing agent in its composition. According to its manufacturer, this sealer has biocompatibility; bioactivity; and high pH, radiopacity, and flow values. However, there is no study in the literature assessing Bio-C sealer.

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Standardized evaluation tests defined by American Dental Association (ADA) specification no. 57[10] and ISO 6876[11] are available to evaluate the physicochemical properties of root canal sealers. However, the conventional methodologies to assess solubility and dimensional stability have some limitations[12,13]. The difference in material weight before and after immersion in water may not represent solubility. Some materials can absorb water even though they present solubility[12]. New methodologies using micro–computed tomographic (micro-CT) imaging have been developed to complement the tests performed by the ADA and ISO[7]. Solubility and dimensional stability can be evaluated similar to clinical conditions by assessing the volumetric change of a material after immersion in a fluid[14,15].

The aim of this study was to evaluate the physicochemical properties of the new Bio-C Sealer in comparison with TotalFill BC calcium silicate–based sealer and the gold standard AH Plus epoxy resin–based sealer using conventional and micro-CT tests. The null hypothesis was that there would be no difference between the physicochemical properties of the new ready-to-use calcium silicate sealer and the already available epoxy resin and silicate-based sealers.

MATERIALS AND METHODS

The endodontic sealers used in this study, their respective manufacturers, and their compositions and proportions are described in Table 1.

Evaluation of Physicochemical Properties

Setting Time

Type IV plaster molds (Durone IV Salmon; Dentsply, Petrópolis, RJ, Brazil) measuring 10 mm in diameter × 1 mm high were manufactured and kept immersed in distilled water for 24 hours at 37°C. Then, the molds were filled with the sealers (*n* = 6). The setting time was evaluated based on ISO 6876:2012 standard[11]. A 100-g Gilmore needle with a 2-mm active tip was placed on the sample surface vertically. The materials were kept in an oven at 37°C and 95% humidity. The setting time was determined starting from the beginning of the manipulation of the sealers up to when the marks of the needles could no longer be observed on the sealer surface.

Flow

The flow test was performed based on ISO 6876:2012 standard[11]. After manipulation of the sealer, 0.05 mL of the material was placed in the center of a glass plate using a graduated disposable syringe (*n* = 10). Next, another glass plate (20 g) was placed over the sealer, and a 100-g load was applied centrally to the top plate for 10 minutes. After this period, the longest diameter and the shortest diameter of the resulting sealer disks were measured using a digital caliper. When a difference of less than 1 mm between the diameters was observed, the mean value was recorded. A second evaluation was made by photographing the material on the plate alongside a millimeter ruler. The images obtained were also evaluated according to Tanomaru-Filho et al[16]. The flow area of the material expressed in mm^2 was obtained using Image Tool 3.0 software (University of Texas Health Science Center at San Antonio, San Antonio, TX).

Radiopacity

Six specimens measuring 10 mm in diameter × 1 mm thick were produced for each group. After the setting process, each sample was positioned on occlusal radiographic films (Insight-Kodak Comp, Rochester, NY) and exposed close to an aluminum scale with variable thickness (from 2–16 mm in 2-mm increments) placed centrally. An X-ray unit (Instrumentarium Dental, Tuusula, Finland) operating at 60 kV, 7 mA, 0.32 pulses per second, and a focus-film distance of 33 cm was used. The films were processed in a standard automatic processor (Dent-X 9000; Dent-X, Elmsford, NY). The radiographs were digitized, and the images were imported into Image Tool 3.0 software. The area of each degree of the aluminum scale and the area of the sealers were selected to determine the radiopacity of the materials, expressed as the equivalent thickness of aluminum (in mm).

pH

The pH values of the sealers were determined by filling polyethylene tubes (Embramed, São Paulo, SP, Brazil) measuring 10 mm high × 1.6 mm in diameter with each material (*n* = 10). Each specimen was placed in a flask containing 10 mL deionized water and stored at 37°C. The pH assessment was performed after 1, 7, 14, and 21 days of immersion. The control was based on the pH values of deionized water in which no samples were immersed. The pH of the solutions was analyzed at each time point using a previously calibrated digital pH meter (Digimed, São Paulo, Brazil). The mean pH of each group in each experimental period was calculated after each measurement (in triplicate).

Solubility

The solubility assessment was performed based on the method developed by Carvalho-Junior et al[17]. Circular plastic molds (*n* = 6) measuring 1.5 mm high × 7.75 mm in diameter were filled with the sealers, and a nylon thread was embedded in the fresh sealer mixture. The samples were placed between 2 glass plates covered with cellophane film. TotalFill BC and Bio-C sealers, which require moisture for setting, were assessed by placing 2 pieces of wet cloth between the mold and the glass plates as described by Tanomaru-Filho et al[7]. This unit was kept at 37°C and relative humidity 3 times longer than the duration of their setting time. The test specimens were removed from the molds, kept in a desiccator, and weighed on a precision balance (Adventurer AR2140; Ohaus Corporation, Parsippany, NJ) until stabilization of the initial mass. Then, they were placed in closed plastic flasks containing 7.5 mL distilled water and kept in an oven at 37°C for 30 days. The nylon threads allowed the sample to be immersed in distilled water without touching the flask walls during the experimental period.

TABLE 1 - Endodontic Sealers, Their Manufacturer, Their Composition, and the Proportion Used

Material	Manufacturer	Composition	Proportion
AH Plus	Dentsply DeTrey GmbH, Konstanz, Germany	Bisphenol-A epoxy resin, bisphenol-F epoxy resin, calcium tungstate, zirconium oxide, silica, iron oxide pigments dibenzyldiamine, aminoadamantane, silicone oil	1 g:1 g (paste/paste)
TotalFill BC	FKG Dentaire SA, La Chaux-de-Fonds, Switzerland	Zirconium oxide, calcium silicates, calcium phosphate monobasic, calcium hydroxide, filler and thickening agents	Ready to use
Bio-C Sealer	Angelus, Londrina, PR, Brazil	Calcium silicates, calcium aluminate, calcium oxide, zirconium oxide, iron oxide, silicon dioxide, dispersing agent	Ready to use

After 30 days, the samples were placed in a desiccator and then reweighed until stabilization of the mass to obtain their final weights. Solubility was obtained by calculating the weight loss after immersion and expressed in percentage terms.

Volumetric Change

Specimens 7.75 mm in diameter × 1.5 mm high ($n = 6$) were prepared and kept in an oven at 37°C and relative humidity for 3 times longer than their setting time. Then, the samples were kept in a desiccator for 24 hours and scanned using a SkyScan 1176 micro-CT scanner (Bruker-MicroCT, Kontich, Belgium). The scanning parameters were as follows: 80 kV voltage, 300 μA current, 18 μm voxel size, copper and aluminum (Cu + Al) filter, and 360° rotation. Afterward, the samples were immersed in plastic flasks containing 7.5 mL distilled water and kept in an oven for 30 days. The position of the specimens in the flask was changed after 15 days to allow contact of both sealer surfaces with the water for the same amount of time. After the experimental period, the samples were placed in a desiccator for 24 hours and scanned again. The reconstruction of the images was performed using NRecon software (V1.6.10.4, Bruker-MicroCT). The correction parameter for smoothing, beam hardening, and ring artifacts was defined for each material. The same parameters were used for the same material at the different time points. The reconstructed images were superimposed on the different time points using the Data Viewer software (V1.5.2.4, Bruker-MicroCT). CTAn software (V1.15.4.0, Bruker-MicroCT) was used for quantitative analysis of the samples, allowing the total volume of material to be calculated in mm^3. The volumetric change between the baseline and the experimental period was then calculated.

Statistical Analysis

The normality of the data was tested using the Shapiro-Wilk test. Statistical analysis was performed with analysis of variance and Tukey parametric tests. The level of significance was set at $P < .05$.

RESULTS

The results are represented in Tables 2 and 3. Bio-C Sealer had the shortest setting time followed by AH Plus and TotalFill BC Sealer ($P < .05$). Bio-C Sealer showed the highest flow, and AH Plus had the lowest in both analyses (mm and mm^2) ($P < .05$). AH Plus showed the highest radiopacity and the lowest solubility and volumetric change ($P < .05$). The solubility rate was higher for Bio-C Sealer ($P < .05$); however, Bio-C Sealer and TotalFill BC Sealer had similar volumetric change ($P > .05$).

The pH was higher for TotalFill BC Sealer followed by Bio-C Sealer after 1 day ($P < .05$). No statistically significant difference was observed between TotalFill BC Sealer and Bio-C Sealer in the other experimental periods ($P > .05$). AH Plus was similar to the control group ($P > .05$).

DISCUSSION

Successful endodontic treatment outcome is achieved by appropriate sealing of the root canal filling materials[5]. A new generation of bioceramic endodontic sealers has been developed using calcium silicate[6]. The main advantages of bioceramic materials are related to their physical and biological properties. Bioceramics are biocompatible, nontoxic, and chemically stable within the biological environment[18].

The physicochemical properties of root canal sealers should be evaluated by means of standardized methodologies defined by ADA standard 57[10] and ISO 6876[11]. The setting time of endodontic sealers should allow enough time for the material to be placed in the root canals[19]. However, a prolonged setting time is considered a critical issue in clinical application[20]. The current study showed that TotalFill BC Sealer had the longest setting time followed by AH Plus, corroborating a previous study[7]. Bio-C Sealer had the shortest setting time.

Flow is an important property for root canal filling[21]. This property allows the sealer to penetrate into the irregularities of root canal systems[22]. All the sealers evaluated presented flow rates in compliance with ISO 6876 standard, corroborating previous studies[7,23]. However, Bio-C Sealer presented the highest flow rate.

The relative radiopacity of root filling materials is essential for assessing the root canal filling[19] and for distinguishing the material from the surrounding anatomic structures[24]. In the present study, the endodontic sealers met the American National Standards Institute/ADA requirements[10]. AH Plus showed the highest radiopacity. TotalFill BC Sealer and Bio-C Sealer presented similar radiopacity. This can most likely be explained by the presence, quantity, and proportion of radiopacifying agents in each material[18]. The radiopacity results of TotalFill BC Sealer and Bio-C Sealer were significantly lower than that of AH Plus, probably because calcium silicate–based sealers have zirconium oxide in their composition, whereas AH Plus contains not only zirconium oxide but also calcium tungstate[18].

Solubility indicates the loss of material mass when immersed in water[19]. Root canal sealers should present solubility less than 3%[10,11] in order to maintain their sealing ability and avoid reinfection[25]. Our results showed that only AH Plus complied with the ADA[10] and ISO standards[11]. The low solubility of AH Plus may be attributed to the strong cross-links in epoxy resin–based materials[26]. On the other hand, calcium silicate–based sealers have shown high solubility after immersion in water compared with the standard resin-based sealers[6]. This high solubility can be explained by the hydrophilic nanosized particles that increase their surface area and allow more liquid molecules to come into contact with the sealer[27].

Although the high solubility of calcium silicate–based sealers can be considered a disadvantage, their bioactive potential is a consequence of the solubility of these materials even after setting[25]. Moreover, the solubility of calcium silicate–based sealers can be explained by the release of OH^- and Ca^{2+} ions[28], thus corroborating our results regarding the alkaline pH for TotalFill BC Sealer and Bio-C Sealer and low solubility and pH values for AH Plus. An alkaline environment may play a positive role in apical healing, thus contributing to the formation of mineralized tissues[29]. The effects of alkaline materials (ie, calcium hydroxide, mineral trioxide aggregate, and other alkaline materials) used as root canal dressings or as filling materials on the

TABLE 2 - Setting Time, Flow, Radiopacity, Solubility, and Volumetric Change Observed in the Different Root Canal Sealers (Mean and Standard Deviation)

Test	AH Plus	TotalFill BC	Bio-C Sealer
Setting time (min)	385.0 (±4.5)[b]	582.2 (±21.5)[a]	220.0 (±12.7)[c]
Flow (mm)	21.3 (±1.1)[c]	24.7 (±0.8)[b]	31.2 (±1.3)[a]
Flow (mm^2)	409.2 (±108.6)[c]	537.4 (±45.0)[b]	868.4 (±34.9)[a]
Radiopacity (mmAl)	9.2 (±0.5)[a]	6.1 (±0.7)[b]	5.5 (±0.6)[b]
Solubility (% mass loss)	0.2 (±0.4)[c]	10.6 (±3.2)[b]	17.9 (±2.5)[a]
Volumetric change (%)	−0.4 (±0.2)[b]	−1.9 (±1.0)[a]	−0.9 (±0.6)[a]

Negative values in the volumetric change test indicate volume loss.
Different letters on the same line indicate statistically significant differences ($P < .05$).

TABLE 3 - pH Values (Mean and Standard Deviation) Observed at the Different Experimental Periods (1, 7, 14, and 21 Days)

Period	AH Plus	TotalFill BC	Bio-C Sealer	Control
1 day	6.66 (±0.24)[c]	10.38 (±0.19)[a]	9.65 (±0.17)[b]	6.51 (±0.32)[c]
7 days	6.12 (±0.37)[b]	10.23 (±0.52)[a]	9.74 (±0.53)[a]	6.53 (±0.30)[b]
14 days	6.53 (±0.37)[b]	10.24 (±0.43)[a]	9.90 (±0.95)[a]	6.52 (±0.27)[b]
21 days	6.23 (±0.24)[b]	9.68 (±0.89)[a]	9.18 (±1.01)[a]	6.43 (±0.38)[b]

Different letters on the same line indicate statistically significant differences ($P < .05$).

mechanical properties of radicular dentin are not conclusive and require further investigation[30]. On the other hand, bioceramic sealers have been associated with the penetration of sealers into dentin tubules[31]. This better penetrability observed for calcium silicate–based sealers may be favorable for root canal sealing[32].

The conventional solubility test may not be appropriate to evaluate materials that absorb water[12,33]. Calcium silicate–based sealers require moisture in order to set and have a reaction to hydration[34]. The solubility of these materials could have been overestimated in the drying process when determining the final mass, considering that the water not incorporated during hydration may evaporate in the desiccator, interfering in the result observed for the real mass loss[33]. It is worth noting that ISO 4049 may be applied to include the procedures for testing both sorption and solubility[35]. Considering that the solubility of materials cannot represent the absence of volumetric stability, methodologies using micro-CT imaging can be applied to complement the evaluation of materials[7,14,15]. Our results showed that TotalFill BC Sealer and Bio-C Sealer had a volumetric change below 2%, even though they showed solubility above 10%. Previous studies also showed low volumetric changes for sealers presenting a large mass loss[7,14]. The volumetric change tests performed using micro-CT imaging can complement the solubility analysis of calcium silicate–based root canal sealers[7] in a more clinical setting[14,15].

Bio-C Sealer is a new sealer available on the market. More research is required before this sealer can be recommended for clinical application. Based on our results, the authors concluded that Bio-C Sealer has a short setting time, alkalinization ability, and adequate flow and radiopacity. Although Bio-C Sealer did not meet the ISO or American National Standards Institute/ADA protocols regarding solubility, this sealer showed low volumetric change.

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

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