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Evaluation of Physicochemical Properties of New Calcium Silicate-Based Sealer

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This study aimed to evaluate the physicochemical properties of a calcium silicate-based sealer (Sealer Plus BC; MK Life, Porto Alegre, Brazil) compared with an epoxy-resin sealer (AH Plus; Dentsply DeTrey GmbH, Konstanz, Germany). Initial and final setting time was assessed based on ISO 6876:2012 and ASTM C266:03. Calcium ion release and pH were evaluated by filling polyethylene tubes with sealers and then immersing them in 10 mL of deionized water. Following experimental periods of 1, 24, 72 and 168 hours, the samples were measured regarding pH and calcium ion release with a pH meter and a colorimetric spectrophotometer, respectively. The flow was examined based on ISO 6876:2012. Rings of 10 mm in diameter with 1 mm thickness were prepared to analyze the radiopacity (ISO 6876:2012 and ADA n.57) and solubility (ISO 6876:2012). The data were analyzed by variance analysis, Student-T and Tukey tests ($p<0.05$). The calcium ion release and pH values were significantly higher for the Sealer Plus BC compared with the AH Plus ($p<0.05$). Lower setting time, flow and radiopacity were observed for the bioceramic sealer than for AH Plus ($p<0.05$). Sealer Plus BC exhibited higher solubility compared with AH Plus ($p<0.05$). Sealer Plus BC showed physicochemical properties as setting time, pH, calcium release, flow, and radiopacity following the required standards, but higher solubility than the minimum values required by ISO 6876:2012.

Key Words: bioceramic sealer, endodontic sealer, calcium silicate-based sealer, physicochemical properties of sealers.

Introduction

Calcium silicate-based sealers, usually known as bioceramic sealers, were introduced to dentistry marketing as an alternative endodontic sealer (1). This material originally came from a combination of calcium silicate and calcium phosphate (2). Calcium phosphate enhances the setting properties of bioceramic sealers resulting in a chemical composition and crystalline structure similar to the tooth and bone apatite materials, thereby improving sealer-to-root dentin bonding (3). A recent systematic review of laboratory research compared the physicochemical and biological properties of bioceramic with those of conventional sealers, and the results were similar or better for the calcium silicate-based sealers (4). The composition of bioceramic sealers might also contain aluminum, zirconia, bioactive glass, glass ceramics and hydroxyapatite (5). This material has an alkaline pH, antimicrobial action, and biocompatibility (6).

Recently, a new calcium silicate-based sealer called Sealer Plus BC (MK Life, Rio Grande do Sul, Brazil) was developed and introduced into the market. This bioceramic sealer is insoluble, radiopaque and did not contain aluminum in its composition. However, it presents calcium silicate, which requires the presence of humidity for the setting process (2). However, up to now, there are no studies in the specific literature about the physicochemical properties of the cited sealer.

AH Plus (Dentsply DeTrey GmbH, Konstanz, Germany) is a resin epoxy-based sealer that is considered to be the gold standard clinical practice due to its physicochemical and biological properties (4,7,8). It presents excellent dimensional stability (9), sealing ability (10), flow (7) and biocompatibility (11).

Therefore, this study aimed to evaluate the physicochemical properties (i.e., setting time, pH and calcium release, flow, radiopacity, solubility) of a new formulation for bioceramic sealers (Sealer Plus BC, MK Life), in comparison to AH Plus sealer (Dentsply DeTrey GmbH). The null hypothesis of this study was that there would be no difference between the sealers related to their physicochemical properties.

Material and Methods

One commercial brand of bioceramic sealer (Sealer Plus BC) and one brand of epoxy resin-based sealer (AH Plus Jet) were tested. AH Plus Jet comes with a mixing tip, which automatically mixes the sealer components in an ideal ratio according to manufacturer's specifications. Sealer Plus BC is a formulation ready for use that must be only pressed using the plunger of the syringe. The quantity of each sealer was dependent on each test. All materials were placed in an analytic scale to measure their weight. Table 1 presents the chemical compositions of the sealers.

Setting Time

The setting time of the sealers was determined according to ISO 6876:2012 (12) and the ASTM C266-03 (ASTM C266-03). Three specimens, measuring 10 mm in diameter and 2 mm in height, were produced for each sealer (n=3). The sealers were manipulated according to the manufacturers' specifications for 120 s and inserted inside the matrix at a temperature of 37 °C and air humidity of 95%. After thirty seconds, a 100 g Gilmore needle with a 2 mm active tip was vertically placed on the sample surface. This procedure was repeated every 60 s until sealer surface was no longer marked, and this point defined the initial setting time. The evaluation of the final setting time began immediately after the initial setting time was determined. At this point, a 456.5 g Gilmore needle with a 1.0 mm active tip was vertically positioned on the sealer surface (13). The same interval of repetitions that was used to determine the initial setting time was applied to determine the final setting time.

pH and Calcium Ion Release

To determine the pH and calcium ion release of Sealer Plus BC and AH Plus sealers, five specimens for each group were produced (n=5) with polyethylene tubes (length of 10 mm and an internal diameter of 1.0 mm) with a closed end (3). The sealers were inserted inside the tubes using 1 mL syringes until they were filled. Each specimen was placed in a flask containing 10 mL deionized water and stored at 37°C. The pH assessment was performed after 1,24,72 and 168 h of immersion. Previously to the readings, the sealer samples were removed from the flasks, and the solutions were agitated for 5 s. The pH was measured with a digital pH meter (Digimed DM-22, São Paulo, SP, Brazil) calibrated with solutions with known pH (4,13). The control for the method was based on the reading of pH values of the deionized water in which no samples were immersed.

The calcium ion release assessment was performed at the same experimental periods used for pH analysis (i.e., 1, 24, 72 and 168 h). The calcium levels contained in the collected samples were examined with a colorimetric method using the arsenazo III (14).

Flow

According to ISO 6876:2012 (12), specimens were produced (n=5) for each sealer. Subsequently, 0.5 ± 0.005 mL sealer was placed on a glass plate with dimensions of 40 mm (height) x 40 mm (width) x 5 mm (thickness) using a 1 mL disposable syringe. Next, another glass plate of the same dimensions was placed over the sealer, and a 100 g load was centrally applied to the material for 10 min. After the force load ceased, the longest and the shortest diameter of the sealer disks produced were measured using a digital caliper (Digimess, São Paulo, SP, Brazil). If the difference between the two diameters was greater than 1 mm, the test was conducted again. The flow values were obtained from the mean values of the three tests performed for each group.

Radiopacity

Following ISO 6876:2012 (12), three specimens were produced for each group. After sealer manipulation, the samples were introduced into a matrix of 10 mm in diameter and 1 mm in height for sample production. After the setting process, the samples were laterally positioned on both sides of a periapical radiographic film close to an aluminum scale that was centrally placed. This scale had a thickness that varied from 0.5 to 5 mm. Next, the radiography procedure used a Timex 70E X-ray device (Saevo, Ribeirão Preto, SP, Brazil) with an exposure time of 0.1 s. Images were digitalized in a Vista Scan Mini Easy system (Durr Dental, Bietigheim-Bissingen, Germany) and analyzed with ImageJ software (Research Services Branch, National Institutes of Mental Health, Bethesda, MD, USA). The grey levels (pixel densities) of the aluminum scale and a 1.5 m^2 standardized area in the center of the samples were calculated regarding their mean values and standard deviations. The radiopacity value was determined according to the radiographic density, which was also converted into millimeters of aluminum (mm Al). The conversion was

Table 1. Chemical compositions of the sealers

AH Plus		Sealer Plus BC
Paste A	Paste B	
Bisphenol-A epoxy resin	Adamantane amine	Calcium silicate
Bisphenol-F epoxy resin	N, N "-Dibenzyl-5-oxanonane diamine-1.9	Zirconium oxide
Calcium tungstate	TCD - Diamina	Tri-calcium silicate
Zirconium oxide	Calcium tungstate	Calcium silicate
Silica	Zirconium oxide	Calcium hydroxide
Iron oxide	Silica	-
-	Silicone oil	-

performed as described by Duarte et al. (15).

Solubility

Three specimens of 10 mm in diameter and 1 mm in height (13) were produced for each group to determine the solubility. After the sealers' setting processes the specimens were removed from the impressions, and every remaining particle on the surface of the disks was removed using a microbrush. The samples were weighed on an analytic scale (Shimadzu, Tokyo, Japan) with a precision of 0.001 g and then placed in Falcon tubes (Labor Import, Osasco, São Paulo, Brazil) with 50 mL distilled water. The tubes were then closed for 7 days (168 h). The specimens were inserted using a nylon thread, which allowed the sample to be hung and immersed in the distilled water without touching the Falcon tube walls for the entire experimental period. After 168 h, the specimens were removed from the tubes, gently washed with distilled water, dried with absorbent papers, placed in a dehumidifier for 24 h and then re-weighed to obtain their final weights. Solubility was obtained by calculating the weight loss after the immersion.

Statistical Analysis

The pH and calcium ion release values were statistically compared using 1-way analysis of variance (ANOVA) and Tukey test. Flow, solubility, radiopacity and initial and final setting times were analyzed using Student-T test. The significance level was set at 5%.

Table 2. Means and statistical comparisons of the pH values, calcium ion release (mg/L), flow (mm), solubility (g), radiopacity (mm/Al) and initial and final setting times (hours and minutes).

	AH Plus	Sealer Plus BC
pH – 1 h	8.95 ^{A} ± 0.16	9.47 ^{B} ± 0.38
pH – 24 h	7.70 ^{A} ± 0.29	9.58 ^{B} ± 0.81
pH – 72 h	7.89 ^{A} ± 0.44	10.05 ^{B} ± 1.17
pH – 168 h	8.17 ^{A} ± 0.32	9.09 ^{B} ± 0.88
Calcium ion release – 1 h	30.01^{A} ± 4.52	196.6 ^{B} ± 33.4
Calcium ion release – 24 h	61.23 ^{A} ± 6.31	379.4 ^{B} ± 33.8
Calcium ion release – 72 h	46.70 ^{A} ± 3.98	392.4 ^{B} ± 10.2
Calcium ion release – 168 h	0.0 ^{A} ± 0.01	340.9 ^{B} ± 43.8
Flow	32,25 ^{A}	29,73 ^{B}
Solubility	0,001 ^{A}	0,017 ^{B}
Radiopacity	8,05 ^{A}	3,67 ^{B}
Initial setting time	10 h 17 min ^{A}	50 min ^{B}
Final setting time	14 h 29 min ^{A}	3 h 13 min ^{B}

Footnote: Uppercase letters denote significant differences in the line. The pH and calcium ion release values were statistically compared using 1-way ANOVA and Tukey test. Flow, solubility, radiopacity and initial and final setting times were compared using Student-T test. The significance level was set at 5%.

Results

Table 2 summarizes the initial and final setting times, pH values, calcium ion release, flow, radiopacity, and solubility. Sealer Plus BC showed higher pH and calcium ion release than AH Plus ($p<0.05$). The setting time, flow and radiopacity values presented by Sealer Plus BC were lower than those obtained with AH Plus ($p<0.05$). Sealer Plus BC had higher solubility than the AH Plus sealer ($p<0.05$). Furthermore, Sealer Plus BC exhibited higher solubility than that recommended by ISO 6876:2012 (12).

Discussion

It is essential to establish standardized methodologies to evaluate physicochemical properties of new and gold standard root canal sealers. This care must be taken to reproduce results and make reliable comparisons of data obtained from several studies (16). The standardization was established after the publication of the American Society for Testing and Materials (17) and ISO 6876:2012 (12).

The setting time should not be too short because that might interfere and complicate filling methods by decreasing the working time. In the present study, 100 and 456.3 g Gilmore needles were used (12,17) to allow the determination of initial and final setting times. Sealer Plus BC presented shorter initial and final setting times than did AH Plus ($p<0.05$). This result can be considered favorable because a slow setting time can result in tissue irritation, once the most root canal sealers produce some degree of toxicity until being completely set (6). And also, it was not too long, therefore impairing its solubilization by the periapical tissues, which would lead to sealing failure. Zhou et al. (18) reported setting time of 2.7 h for a bioceramic sealer (i.e., Endosequence; Brasseler, Savannah, GA, USA) similar to that used in this study. According to Loushine et al. (2) the bioceramic sealers present two phases of reaction. In the first phase, monobasic calcium phosphate reacts with calcium hydroxide in the presence of water and produce water and hydroxyapatite. In the second phase, the water derived from the dentin humidity, as well as that from the phase I reaction, contributes to the hydration of calcium silicate particles to trigger a calcium silicate hydrate phase (2).

High pH values assume particular importance for root canal sealers because the releasing of calcium ions stimulates the deposition of hard tissue and their antibacterial properties (6). In the present study, the tri-calcium silicate-based sealer exhibited significantly higher pH values than the epoxy resin-based sealer in all experimental periods ($p<0.05$). Similarly, Zhou et al. (18) found lower pH values for the AH Plus in comparison with two bioceramic sealers up to 800 h of evaluation. Sealer Plus BC presented an alkaline pH (ranging from 9.09 to 10.05) that remained stable for 168 h (7 days). The alkalizing effect can be explained by the presence of calcium hydroxide in the matrix composition of this material. Accordingly, the results for the pH values for the bioceramic sealer and AH Plus in this study were similar to those reported by Khalil et al. (7).

The calcium ion release and, in consequence, the increase in pH values is closely related to setting time and solubility (19). According to Parirock and Torabinejad (20), the presence of calcium may favor an alkaline pH, which leads to a biochemical effect that accelerates the healing process. The high pH values found with Sealer Plus BC can be explained because of its high solubility despite the lower setting time when compared with AH Plus. This study used the colorimetric method to evaluate this outcome. When Ca^{++} values are extremely low, the results of this method are expressed in values close to zero, which justified the results obtained in 168 h for AH Plus. Other studies have also found higher levels of calcium ion release for bioceramic sealers compared with others sealers, even including AH Plus (3,21).

The flow of Sealer Plus BC was slightly lower than that of AH Plus ($p<0.05$), but both of the sealers met the ISO 6876:2012 (12) specification which requires a flow rate not less than 20 mm. Although good flow contributes to good penetration into the dentine tubules, isthmus, accessory canals, it is essential to emphasize that excessive flow might cause leaks into the periapical tissues, which, depending on how cytotoxic the material is, might complicate the healing process (15). Furthermore, some authors highlight that it is not crucial that the flow of bioactive sealers are in agreement with the ISO regulations (7,10). This thought is based on their bioactivity, once mineral infiltration zone is formed, and the sealer becomes part of the dentine (10). Thus, the thicker film thickness may not impair the sealing ability of root canal fillings performed using bioactive sealers (7). The flow rate found in this study for Sealer Plus BC is in agreement with those obtained with other bioceramic sealers which ranged between 23.1 mm and 29 mm (7,18).

Radiopacity is an essential property of root canal sealers because it allows the visualization of the quality of the filling content. Moreover, endodontic sealers should be sufficiently radiopaque to be distinguishable from adjacent anatomical structures (6). According to international standards, endodontic sealers must present a minimum radiopacity equal to 3.00 mm of aluminum (22). Comparison of the radiopacities of the two sealers revealed that Sealer Plus BC (3.67 mmAl) was less radiopaque than AH Plus (8.05 mmAl) ($p<0.05$). In agreement with the present study, previous authors have also reported lower radiopacity values for bioceramic sealers than for AH Plus (3,7). Such result occurred due to the radipacifiers present in each sealer. Sealer Plus present only zirconium oxide; while AH Plus combines zirconium oxide and calcium tungstate. EndoSequence BC Sealer, in turn, presents similar radiopacity values with Sealer Plus BC (3.83 mmAl) because also contains zirconium oxide in its chemical composition (3).

Solubility is straightforwardly related to the dissociation of material components by contact actions with surrounding fluids, creating gaps that could be colonized by microorganisms and lead to reinfection (23). In this study, the solubility of Sealer Plus BC was higher than that of AH Plus ($p<0.05$), and did not meet the minimum requirements of ISO 6876:2012 (12), which claims that the loss of mass must not exceed 3% of the total weight. A possible explanation for this observation might be the fact the characteristics of hydrophilic materials may be altered due to humidity (6). Consequently, it might be inferred that, clinically, tissue fluids and blood do not wait until the setting process is complete to wet the material (23). Therefore, the solubility values are probably even higher under clinical conditions (24). As observed in the present study, Zhou et al. (18) also demonstrated that their tested bioceramic sealer (Endosequence BC Sealer) exhibited higher solubility values than their other tested materials, which included AH Plus. Another bioceramic sealer also presented high solubility values (iRoot SP; Innovative BioCeramix Inc., Vancouver, Canada), close to 20% of the loss of mass. The high solubility of bioceramic sealers occurs as the result of hydrophilic nanosized particles which increases their surface area and allows more liquid molecules to come into contact with the sealer (6).

Sealer Plus BC is a new sealer available in the market, and present no parameters for comparison. More investigations are needed for extensive clinical use. Based on the methodology employed and the results obtained, Sealer Plus BC exhibited excellent physicochemical properties, such as pH, calcium ion release, flow, radiopacity and setting time. However, this new bioceramic sealer showed higher solubility than recommended by ISO 6876:2012 (12).

Resumo

Este estudo teve por objetivo avaliar as propriedades físico-químicas de um cimento à base de silicato de cálcio (Sealer Plus BC MK Life, Porto Alegre, Brasil) e compará-las a um cimento à base de resina epóxica (AH Plus, Dentsply DeTrey GmbH, Konstanz, Alemanha). Tempo de presa inicial e final foram avaliados com base na ISO 6876:2012 e ASTM C266:03. Liberação de íons cálcio e pH foram avaliados após o preenchimento de tubos de polietileno com os cimentos e imersão em

10 mL de água deionizada. Após os tempos experimentais de 1,24,72 e 168 horas, os valores de pH e liberação de íons cálcio foram mensurados utilizando um medidor de pH e um espectofotômetro colorimétrico, respectivamente. Escoamento foi avaliado com base na ISO 6876:2012. Moldes de 10 mm de diâmetro e 1 mm de espessura foram preparados para análise de radiopacidade (ISO 6876:2012 e ADA n.57), solubilidade (ISO 6876:2012). Os dados foram analisados por análise de variância, teste T de Student e teste de Tukey (p<0,05). A liberação de íons cálcio e os valores de pH foram significativamente maiores para o Sealer Plus BC em comparação com o AH Plus (p<0,05). Menores valores de tempo de presa, escoamento e radiopacidade foram observados para o cimento biocerâmico quando comparados com o AH Plus (p<0,05). Sealer Plus BC apresentou propriedades físico-químicas de tempo de presa, pH, liberação de íons cálcio, escoamento, radiopacidade de acordo com as normas exigidas, porém maior solubilidade que aquelas previstas pela ISO 6876:2012.

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