

Research Article



Flow characteristics and alkalinity of novel bioceramic root canal sealers

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Conflict of Interest

No potential conflict of interest relevant to this article was reported.

Author Contributions

Conceptualization: Katakidis A, Koulaouzidou E, Economides N; Data curation: Katakidis A, Gogos C; Formal analysis: Katakidis A, Sidiropoulos K, Koulaouzidou E, Gogos C, Economides N; Investigation: Katakidis A, Koulaouzidou E, Economides N; Methodology: Katakidis A, Koulaouzidou E; Project administration: Koulaouzidou E, Economides N; Software: Katakidis A, Sidiropoulos K, Gogos

ABSTRACT

Objective: This study aimed to examine the physical properties (pH and flow) of 2 novel bioceramic sealers.

Materials and Methods: The tested sealers were a calcium hydroxide sealer (Sealapex) and 2 bioceramic sealers (BioRoot RCS and TotalFill BC Sealer). Flow measurements were conducted according to ISO 6876/2012, with a press method of 0.05 mL of sealer. The pH of fresh samples was tested immediately after manipulation, while set samples were stored for 3 times the recommended setting time. The predetermined time intervals ranged from 3 minutes to 24 hours for fresh samples and from 10 minutes to 7 days and 4 weeks for the set samples. Analysis of variance was performed, with $p = 0.05$ considered indicating significance.

Results: The mean flow values were 26.99 mm for BioRoot, 28.19 for Sealapex, and 30.8 mm for TotalFill BC Sealer, satisfying the ISO standard. In the set samples, BioRoot RCS had higher pH values at 24 hours to 1 week after immersion in distilled water. At 2 weeks, both bioceramic sealers had similar pH values, greater than that of Sealapex. In the fresh samples, the bioceramic sealers had significantly higher initial pH values than Sealapex ($p < 0.05$). At 24 hours post-immersion, all sealers showed an alkaline pH, with the highest pH observed for TotalFill.

Conclusions: The TotalFill BC Sealer demonstrated the highest flow. The bioceramic sealers initially presented higher alkaline activity than the polymeric calcium hydroxide sealer. However, at 3 and 4 weeks post-immersion, all sealers had similar pH values.

Keywords: Bioceramics; Flow; Alkalinity; Root canal sealers





INTRODUCTION

Endodontic sealers, in combination with gutta-percha, play a vital role in root canal obturation, and therefore for the success of endodontic treatment in general [1]. The ideal properties of a sealer include adhesion with the canal wall, slow setting, no shrinkage, insolubility in tissue fluids, biocompatibility, and bactericidal ability [2,3].

A wide range of endodontic sealers has been used in clinical practice, including zinc oxide-eugenol sealers, resin sealers, calcium hydroxide sealers, and recently developed bioceramic

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sealers. Bioceramic sealers have recently attracted considerable attention due to their physical and biological properties [4]. They have shown great potential in terms of biocompatibility and adhesion with canal walls due to their crystalline structure, which is very similar to that of the tooth [5].

A novel bioceramic root canal sealer, BioRoot RCS (Septodont, Louisville, KY, USA), has been reported to exhibit strong and prolonged alkaline activity, calcium release, and apatite-forming ability [6]. According to the manufacturer's information, it consists of a powder primarily composed of tricalcium silicate and zirconium oxide, while its liquid component is an aqueous solution of calcium chloride. Another novel bioceramic root canal sealer, TotalFill BC Sealer (BUSA, Savannah, GA, USA), was recently developed and is mainly composed of zirconium oxide, tricalcium silicate, dicalcium silicate, and calcium hydroxide according to the manufacturer's safety data sheet.

An alkaline pH is associated with bactericidal and bacteriostatic properties and deposition of mineralized tissue. Therefore, the maintenance of an alkaline pH for an extended period after treatment is an important property of endodontic sealers. Calcium hydroxide root canal sealers were developed to incorporate the beneficial properties of calcium hydroxide in terms of alkaline pH and calcium ion release. A study showed that Sealapex (Kerr, Orange, CA, USA) was the calcium hydroxide root canal sealer with the highest pH and calcium ion release [7]. According to the manufacturer's safety data sheet, its main ingredients are N-ethyl-o(or p)-toluenesulfonamide (10%–30%), calcium oxide (10%–30%), zinc oxide (1%–5%), and isobutyl salicylate (1%–5%). Only a few studies have investigated the pH changes of BioRoot RCS and TotalFill BC Sealer. The pH of BioRoot RCS has been shown to increase for a prolonged period of time [6,8], but the pH change of TotalFill BC Sealer has only been investigated over a short period [9,10].

The flow rate of the sealer is another important factor that affects the outcome of the final root filling. A sealer with a high flow rate may be capable of filling canal irregularities, accessory canals, and isthmuses [6]. However, as the flow rate increases, the risk of sealer extrusion beyond the apical foramen becomes more likely, which can lead to various tissue responses, ranging from delayed tissue repair to foreign body reaction [10]. To our knowledge, this is one of the few studies to examine the flow rate of BioRoot RCS and TotalFill BC Sealer.

Hence, the aim of the present study was to evaluate the physicochemical properties of 2 novel bioceramic root canal sealers in comparison with a calcium hydroxide sealer. In particular, the pH changes of the fresh and set sealers, as well as the flow rate, were investigated.

MATERIALS AND METHODS

Two novel bioceramic root canal sealers (BioRoot RCS and TotalFill BC Sealer) and a polymeric calcium hydroxide sealer (Sealapex) were used as experimental materials. BioRoot RCS and Sealapex were mixed and handled according to the manufacturer's instructions. TotalFill BC Sealer is a premixed material, making further manipulation unnecessary. The physical property of flow was tested according to the ISO 6876:2012 specifications [11], and pH values were measured at different time intervals in fresh and set samples.

Flow measurements

The flow of the sealers was tested according to ISO 6876:2012. A volume of 0.05 ± 0.005 mL of mixed sealer was prepared and placed on the center of a glass plate ($40 \times 40 \times 5$ mm³) using a disposable 1.0 mL syringe. At 180 ± 5 seconds after the commencement of mixing, a second glass plate weighing 20 g with the same dimensions as the initial one was placed centrally on top of the sealer and a total mass of 120 g was applied on the first plate. Ten minutes after the onset of mixing, the weight was removed, and the minimum and maximum diameters of the compressed sealer discs were measured using a digital caliper (Cole-Parmer Canada Inc., Montreal, Canada) with a resolution of 0.01 mm [12]. If the diameters were within 1 mm of each other, the mean value of both was taken as the sample flow. If the 2 diameters were not within 1 mm of each other or uniformly circular, the test was repeated. Five samples of each sealer ($n = 5$) were prepared, and the mean value of 3 measurements for each sample, rounded to the nearest millimeter, was considered to be the flow of the material. In agreement with the ISO 6876:2012 specifications for the flow test, disks with a diameter smaller than 20 mm were rejected, and the test was repeated in such cases.

Alkalinity evaluation

Fresh and set samples of the root canal sealers were tested.

Fresh samples were tested immediately after mixing. Three samples were prepared for each sealer. A volume of 0.02 mL of each fresh sample was placed in a flat-bottom test glass tube containing 10 mL of distilled water. The samples were stored in an incubator (37°C, > 95% relative humidity) for various time intervals (3, 20, 60, and 120 minutes and 24 hours).

Set samples were prepared as follows ($n = 3$ for each sealer). The root canal sealer was manipulated and injected into a silicone rubber mold with a diameter of 5 mm and a height of 1 mm. Then, the samples were stored in an incubator (37°C, > 95% relative humidity) for a period equal to 3 times the final setting time according to the manufacturer's instructions. BioRoot RCS and TotalFill BC Sealer samples were stored for 12 hours and Sealapex samples were stored for 135 minutes. Then, each set sample was immersed in a test glass tube containing 10 mL of distilled water, which was sealed with laboratory film (Parafilm "M", Bemis, Neenah, WI, USA). The samples were stored in the incubator (37°C, > 95% relative humidity) for various time intervals (10, 30, 60, 120, 180, 240 minutes, 1, 2, 4, 5, 6, 7 days, and 2, 3, 4 weeks).

The pH values were calculated with a digital pH meter (Thermo Scientific Orion 3-Star Benchtop pH Meter, Thermo Scientific, Waltham, MA, USA) [13,14], previously calibrated with buffer solutions of known pH (pH = 4.00, 7.00, 10.00). Prior to the immersion of specimens, the pH of distilled water was measured, and a pH value of 5.5 was obtained. The distilled water was not changed throughout the testing period. After the predetermined time intervals, the specimens were removed, and the test tubes were shaken for 5 seconds before pH measurement. Ten measurements were made for each sample and condition at room temperature (25°C). The electrode of the pH meter was rinsed with distilled water before conducting each measurement in order to eliminate any possible interference [8].

Statistical analysis

The mean and standard deviation of flow and pH for each sealer were calculated and data were statistically analyzed using SPSS version 25 (IBM Corp., Armonk, NY, USA). Statistical analysis was performed by analysis of variance and the *post hoc* test at the level of significance of $p = 0.05$.

RESULTS

Flow analysis showed that all studied root canal sealers demonstrated a flow value greater than 20 mm, satisfying the ISO 6876:2012 recommendations [11]. The mean flow values \pm standard deviations of BioRoot RCS, TotalFill BC Sealer, and Sealapex were 26.99 ± 1.23 mm, 30.8 ± 0.32 mm, and 28.19 ± 1.43 mm, respectively. TotalFill BC Sealer demonstrated a significantly higher flow rate than BioRoot RCS and Sealapex ($p < 0.05$). The flow test results of TotalFill BC Sealer and Sealapex were not significantly different.

The mean pH values of the set and fresh samples are listed in **Tables 1** and **2**, respectively. In the set samples, the bioceramic root canal sealers presented an alkaline pH at all time points, with the maximum pH value recorded at 2 days. Sealapex showed a slightly alkaline pH throughout the observation period (**Figure 1**). In the fresh samples, the initial pH value of the bioceramic sealers was significantly higher ($p < 0.05$) than that of Sealapex. At 24 hours after immersion, all root canal sealers showed an alkaline pH, and TotalFill BC Sealer showed the highest pH (**Figure 2**).

Overall, the pH of the bioceramic root canal sealers was significantly higher than that of Sealapex ($p < 0.05$). In the set samples, BioRoot RCS presented higher pH values than TotalFill BC Sealer and Sealapex at 24 hours and 2, 4, 5, 6, and 7 days after immersion. At 2 days after immersion, BioRoot RCS presented the highest pH value (11.85). At 2 weeks after immersion, both bioceramic sealers presented similar pH values, which were higher than that of Sealapex. At 3 and 4 weeks after immersion, all sealers showed a stable alkaline pH (around 8). In the fresh samples, the initial pH values of BioRoot RCS and TotalFill BC Sealer (10.43 and 10.55, respectively) were significantly higher ($p < 0.05$) than that of Sealapex (5.75). At 24 hours after immersion, all root canal sealers showed an alkaline pH, with TotalFill BC Sealer showing the highest pH (12.14).

DISCUSSION

Flow rate is an important property of endodontic sealers, as it reflects their ability to fill irregularities and voids in the root canal system in order to achieve a hermetic seal [15]. Although a high flow rate is desirable, beyond a certain point, it may contribute to extrusion of sealer beyond the apex to periapical tissues with negative effects on the outcomes of root canal treatment [16,17]. In contrast, a low flow rate affects the handling properties of the

Table 1. Mean values and standard deviation of pH values of set samples

Materials	10 min	30 min	1 hr	2 hr	3 hr	4 hr	24 hr	2 day	4 day	5 day	6 day	7 day	2 wk	3 wk	4 wk
Sealapex	7.01 ^{aA}	8.8 ^{bA}	9.5 ^{bA}	9.9 ^{bA}	10 ^{bA}	10.1 ^{bA}	10.1 ^{bA}	10.1 ^{bA}	9.9 ^{bA}	9.4 ^{bA}	9.5 ^{bA}	8.9 ^{bA}	7.9 ^{aA}	8.3 ^{aA}	7.9 ^{aA}
BioRoot	10.5 ^{aB}	11.1 ^{bB}	11.2 ^{bB}	11.4 ^{bB}	11.4 ^{bB}	11.5 ^{bB}	11.8 ^{bB}	11.6 ^{bB}	11.6 ^{bB}	11.2 ^{bB}	10.6 ^{aB}	10.1 ^{bB}	8.5 ^{cB}	8.3 ^{cA}	8.3 ^{cA}
TotalFill	10.5 ^{aB}	11.1 ^{bB}	11.4 ^{bB}	11.3 ^{bB}	11.3 ^{bB}	11.2 ^{bB}	10.5 ^{aA}	11.3 ^{bB}	10.1 ^{aA}	9.9 ^{aA}	9.5 ^{aA}	9.3 ^{aA}	8.5 ^{cB}	8.2 ^{cA}	7.9 ^{cA}

Different small letters in a horizontal line represent statistically significant differences ($p < 0.05$). Different capital letters in superscript represent statistically significant differences ($p < 0.05$) among materials.

Table 2. Mean values and standard deviation of pH values of fresh samples

Materials	3 min	20 min	1 hr	2 hr	24 hr
Sealapex	5.45 ^{aA}	5.75 ^{aA}	6.39 ^{bA}	8.32 ^{cA}	10.71 ^{dA}
BioRoot	9.98 ^{aB}	10.43 ^{bB}	10.77 ^{bB}	11.31 ^{cB}	11.49 ^{cB}
TotalFill	8.23 ^{aC}	10.55 ^{bB}	10.84 ^{bB}	11.03 ^{bB}	12.14 ^{cC}

Different superscript small letters in a horizontal line represent a statistically significant difference ($p < 0.05$). Different superscript capital letters represent a statistically significant difference among materials ($p < 0.05$).



Figure 1. pH measurements in set samples.

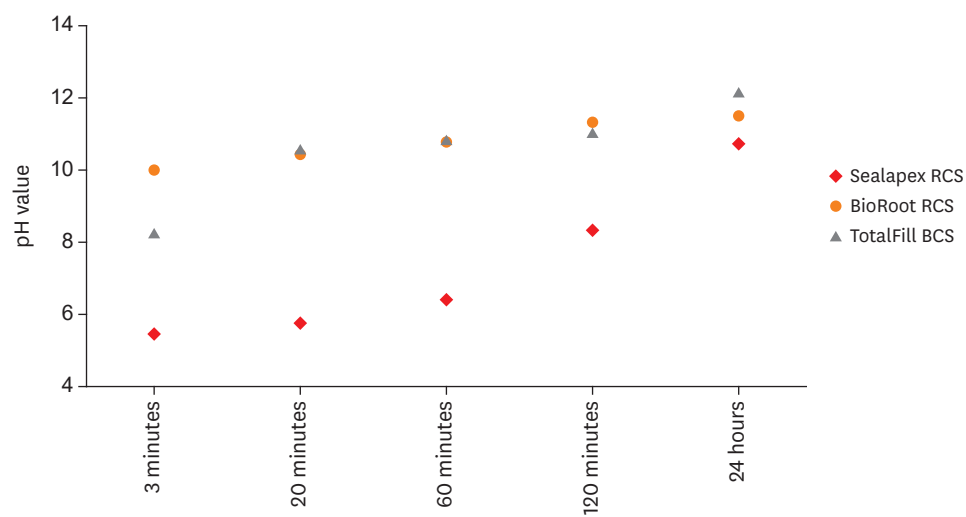


Figure 2. pH measurements in fresh samples.

root canal sealer and could lead to inadequate obturation of the root canal system. This issue demonstrates the necessity of investigating the maximum required values of the flow rate for a sealer. The flow rate was tested according to ISO 6876:2012. In the present study, BioRoot RCS, TotalFill BC Sealer, and Sealapex presented higher flow rates than the minimum required by the ISO standard. The synthesis, filler size, shear rate, temperature, filling technique, and working time are of major significance regarding the flow property of sealers [12,18]. In general, resin-based materials have higher flowability than bioceramic-based materials. The reported values for a typical resin-based sealer, AH-Plus, range from 21.87 mm to 32.25 mm [13,18,19].

In this study, the flow rate of TotalFill BC Sealer was significantly higher than that of BioRoot RCS ($p < 0.05$). Few studies have analyzed the flow rate of BioRoot RCS and TotalFill BC Sealer according to ISO 6876:2012, and limited results are therefore available to compare with our study. The flow rate of BioRoot RCS observed in the present study does not agree with the mean value of 16 mm reported in the literature, which is below the minimum required by

the international standard [20]. Regarding TotalFill, Tanomaru-Filho *et al.* [21], found a flow rate (24.83 mm) lower than that observed in this study, but above the minimum required. For Sealapex, the flow rate found in our study is higher than those given in the literature (*e.g.*, 22.4 mm and 19 mm) [22,23] and in accordance with the international standard. In 1 previous study, the flow rate of Sealapex did not correspond to the required international standard [23].

Another bioceramic root canal sealer, Endosequence BC, demonstrated a high flow value, in agreement with ISO 6786/2012 recommendations [15]. In another study, though, that sealer did not comply with the ISO 6786/2012 recommendations [18]. Other bioceramic-based sealers exhibited flow values ranging from borderline acceptable, such as EndoSeal MTA, to relatively high, as observed for MTA Fillapex [18,24]. The high flow value of MTA Fillapex is attributed to the high amount of resin in its composition [18,25].

The flow properties of endodontic sealers can either be tested with ISO 6876:2012 or the American Dental Association (ADA) specification No. 57 [26]. The main difference between ADA and ISO standards is that the volume of sealer used to measure flow values is 0.5 mL and 0.05 mL, respectively. Both standards set the minimum acceptable flow value as ≥ 20 mm. Some studies have argued that according to the ADA standard, the minimum acceptable value should be ≥ 25 mm [23]. The amount of sealer used could indeed alter the flowability of the material, assuming that the other parameters (such as the mass load, size of the plate, and time) are the same. This might explain the higher flow values obtained using the ADA standard in other studies [23,27].

The pH of distilled water was 5.5, indicating that it was acidic. In another study, the distilled water used as control had a pH value of 5.6 [19]. Pure distilled water has a pH of 7 at 25°C. However, when in contact with the atmosphere, carbon dioxide is absorbed and the pH falls [28]. In other studies, the pH values of distilled water ranged from 6.5 to 6.9, which is considered to be neutral [15,24]. It may be hypothesized that these discrepancies among studies in the pH values of distilled water could explain the differences in the pH values reported for materials in contemporary literature.

Conflicting results have been reported in the literature regarding the pH values of BioRoot RCS after 1 month of immersion. In particular, the reported pH values of set samples range from 8.4 [6], which is consistent with our study, to 12.7 [8,20]. According to 1 study [8], BioRoot RCS presented an alkaline pH in artificial saliva after 1 month of immersion. The pH values observed in our study in the fresh samples at 3 and 24 hours of immersion are in agreement with those in the literature [6].

According to the literature, the TotalFill BC Sealer showed similar pH values in fresh samples at 3 hours [29] and at 1 day [21,29] of immersion, in accordance with those exhibited in our study. At 1 month, the specimens that were immediately immersed after manipulation presented pH values close to 9.23 [21,29]. In the current study, although the pH values were lower than those mentioned above, they were obtained for set samples of TotalFill BC Sealer. For set samples in this study, the pH at 3 hours was higher than that reported in another study, whereas at 1 day after immersion, the recorded values were comparable [9].

The pH values of Sealapex in fresh samples ranged from 9.72 at 24 hours to 10.99 at 1 month [30], whereas another *in vitro* study [31] reported a lower pH value at 24 hours. In contrast, the pH in this study was more basic at 24 hours, close to 10.5. Another study [32] that assessed

the pH level of Sealapex concluded that after 1 week of immersion in distilled water, the mean pH was highly alkaline. In the set samples, the pH values recorded at 3 hours and 24 hours were alkaline, close to 9.7 [9]. These values correspond to those found in our study. Another study [33] reported that the pH values at 5 to 15 hours after immersion were between 10.4 and 10.6. These results agree with our study, as the set samples showed similar pH values for the corresponding time intervals. It can be inferred that the above-mentioned studies agree with the present study regarding the time-related increase of pH level observed for Sealapex.

The differences among BioRoot RCS, TotalFill BC Sealer, and Sealapex may be attributed to the fact that BioRoot RCS and TotalFill BC Sealer are pure bioceramic sealers, while Sealapex is a traditional polymer calcium hydroxide sealer. Calcium silicate-based materials are known to have a high alkalizing ability as a result of the hydration process [34]. A high (alkaline) pH value is of major significance in terms of antibacterial ability, biocompatibility, and osteogenic capacity [12,35-37]. Alkaline materials, with a high pH, are likely able to neutralize the lactic acid secreted from osteoclasts, thereby preventing the absorption of mineralized tooth structure. In this way, hard tissue formation is induced by the activation of alkaline phosphates, favoring the healing process of periapical tissues in general [38].

CONCLUSIONS

Considering the limitations of this *in vitro* study, we conclude that the flow values of all studied root canal sealers were in accordance with the ISO 6876:2012 recommendations. TotalFill BC Sealer had the highest flow, indicating that it likely has a superior ability to penetrate effectively into root canal irregularities. Bioceramic sealers initially presented higher alkaline activity than the polymeric calcium hydroxide sealer, but at 3 and 4 weeks after immersion, all sealers demonstrated similar pH values.

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