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Journal of Dental Sciences xxx (xxxx) xxx

Available online at www.sciencedirect.com
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Original Article

Physicochemical properties of a novel bioceramic silicone-based root canal sealer

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Received 4 September 2021; Final revision received 24 September 2021
Available online ■ ■ ■

KEYWORDS
Differential scanning calorimetry;
GuttaFlow bioseal;
Physicochemical properties;
Root canal sealer

Background/purpose: With introduction into endodontics, bioceramic-based sealers have gained considerable popularity for excellent properties. The aim of this study was to investigate the physicochemical properties of a novel bioceramic silicone-based sealer, GuttaFlow Bioseal, and measure heat flow of setting reactions.

Materials and methods: Film thickness, flow, working and setting time of Bioseal were compared with other 4 kinds of sealers: iRoot SP, AH Plus, RoekoSeal and GuttaFlow2. Differential scanning calorimetry test was performed to measure heat flow.

Results: Bioseal demonstrated the highest film thickness of 44 μm, double to triple that of the other 4 sealers ($P < 0.05$). The highest flow was detected in iRoot SP and RoekoSeal, with values of 27.35 and 27.20 mm, while GuttaFlow2 and Bioseal had the lowest of 22.31 and 21.43 mm ($P < 0.05$). For each sealer, working time at 37 °C was shorter than that at 23 °C ($P < 0.05$). At 37 °C, Bioseal had the shortest working and setting time of 4.5 and 16.3 min, while iRoot SP showed the longest of 105.0 and 571.7 min ($P < 0.05$). Differential scanning calorimetry test revealed that setting process of all the tested sealers was exothermic. Bioseal reached an exothermic peak at 14 min, with almost 1.5 times peak intensity of GuttaFlow2 and RoekoSeal. Whereas iRoot SP and AH Plus reached an exothermic peak 5 h after mixing, with intensity 1/2 to 2/3 that of Bioseal.

Conclusion: The novel bioceramic silicone-based sealer Bioseal showed intense and fast exothermic reactions with characteristic physicochemical properties.

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https://doi.org/10.1016/j.jds.2021.09.034

Please cite this article as: W.-J. Lyu, W. Bai, X.-Y. Wang et al., Physicochemical properties of a novel bioceramic silicone-based root canal sealer, Journal of Dental Sciences, https://doi.org/10.1016/j.jds.2021.09.034

Introduction

A root canal sealer is utilized to seal the space between the dentinal wall and the core material, gutta-percha, as well as fill the anatomical irregularities, lateral and accessory canals. Grossman outlined the properties of an ideal sealer, such as sufficient working time, dimensional stability, biocompatibility and bacteriostatic properties.[1] However, to date, no sealer satisfies all the criteria mentioned above.

With introduction into endodontics, bioceramic-based sealers have gained considerable popularity for excellent physicochemical and biological properties. Bioceramic-based sealers are bioactive due to the ability to form hydroxyapatite on the sealer surface when in contact with tissue fluids.[2,3] GuttaFlow Bioseal, a novel bioceramic silicone-based sealer, contains bioceramic particles and gutta-percha powder in a silicone matrix. Studies have evaluated the bioactivity of Bioseal and its insolubility to tissue fluids, which represents an attractive strategy.[4,5] However, the influence of multibased components on setting reactions for Bioseal has not been studied adequately, and there are limited literatures about the clinically relevant physicochemical properties of Bioseal.[6,7]

The aim of this study was to evaluate the physicochemical properties of a novel bioceramic silicone-based sealer Bioseal, and to investigate the characteristics of its setting reactions. Bioseal, which contains bioceramic particles $CaO-SiO_2-Na_2O-ZrO_2-P_2O_5$ in the range of 20–40 μm, was developed on the basis of silicone-based sealer RoekoSeal and flowable gutta-percha system GuttaFlow2. In the present study, clinically commonly used epoxy resin-based sealer AH Plus and popular bioceramic-based sealer iRoot SP were also compared with Bioseal.

Materials and methods

The composition of the 5 tested sealers in this study is presented in Table 1. The sealers were manipulated according to the manufacturers' instructions.

Physicochemical properties: conventional methods

Film thickness, flow, working and setting time were examined according to ISO 6876 (2012) specifications and tested in triplicate.[8]

Film thickness

According to ISO 6876 (2012) specifications, a volume of 0.05 mL of each sealer was dispensed between two glass plates ($200\ mm^2 \times 5$ mm) using a graduated syringe. After 180 ± 10 s from the start of mixing, a load of 150 N was applied vertically on the plates by a loading device (ELE International, Leighton Buzzard, UK). After 10 min from the start of mixing, the combined thickness of the two plates was measured using a micrometer (Mitutoyo Corporation, Kanagawa, Japan) to an accuracy of 1 μm. The difference in thickness between the two plates with and without sealer was recorded as film thickness.

Flow

According to ISO 6876 (2012) specifications, a volume of 0.05 mL of each sealer was dispensed on the center of a glass plate (40 mm × 40 mm × 5 mm) using a graduated syringe. After 180 ± 5 s from the start of mixing, another glass plate weighing 20 g and a 100-g weight were carefully placed on the top of the sealer. After 10 min from the start of mixing, the maximum and minimum diameters of the compressed disc of sealer were measured using a digital caliper (Mitutoyo Corporation) to an accuracy of 0.01 mm. The mean diameter was calculated and recorded as flow if the diameters agreed to within 1 mm. If not, the test was repeated.

Working time

Working time test was performed following the same procedure as the flow test. It was repeated with freshly mixed material at increasing time intervals from sealer mixing to the time the second glass plate was placed. Each time the test was performed, the maximum and minimum diameters

Table 1 Manufacturer and composition of the 5 tested sealers.

Sealer	Manufacturer	Composition
Bioseal	Coltene/Whaledent, Langenau, Switzerland	Gutta-percha powder, polydimethylsiloxane, platinum catalyst, zirconium dioxide, silver (preservative), coloring, bioactive glass ceramic
iRoot SP	Innovative BioCeramix, Vancouver, Canada	Zirconium oxide, calcium silicates, calcium phosphate monobasic, calcium hydroxide, filler and thickening agents
AH Plus	Dentsply DeTrey, Konstanz, Germany	Bisphenol A/F epoxy resin, calcium tungstate, zirconium oxide, silica, iron oxide pigments, dibenzyldiamine, aminoadamantane, silicone oil
RoekoSeal	Coltene/Whaledent, Langenau, Switzerland	Polydimethylsiloxane, silicone oil, paraffin oil, platinum catalyst, zirconium dioxide
GuttaFlow2	Coltene/Whaledent, Langenau, Switzerland	Gutta-percha powder, polydimethylsiloxane, platinum catalyst, zirconium dioxide, microsilver (preservative), coloring

Table 2 Film thickness and flow of the 5 tested sealers (mean ± standard deviation).

	Bioseal	iRoot SP	AH Plus	RoekoSeal	GuttaFlow2
Film thickness (μm)	44±1[a]	13±1[d]	18±3[bc]	17±1[c]	21±2[b]
Flow (mm)	21.43 ± 0.08[c]	27.35 ± 0.29[a]	26.16 ± 0.28[b]	27.20 ± 1.01[a]	22.31 ± 0.37[c]

[a,b,c,d] Different letters in the same row indicate statistically significant differences ($P < 0.05$).

of the compressed disc of sealer were measured using a digital caliper (Mitutoyo Corporation). The time interval was recorded as working time when the mean diameter decreased to 17 mm.

Setting time

According to ISO 6876 (2012) specifications, stainless steel molds measuring 10 mm in diameter with a height of 2 mm were prepared and filled with AH Plus, RoekoSeal and GuttaFlow2. Because moisture is needed for setting, different molds were used for Bioseal and iRoot SP. Gypsum molds measuring 10 mm in diameter with a height of 1 mm were prepared and stored in an incubator (Shanghai Yiheng Technology Co., Ltd., Shanghai, China) at 37 °C (95% humidity) for 24 h, then filled with Bioseal and iRoot SP respectively. All the molds were kept in the incubator at 37 °C (95% humidity). A 100-g Gilmore needle with a flat end of 2 mm in diameter was placed on the sealer surface vertically. The time from sealer mixing to the point at which the needle failed to make a visible indentation was recorded as setting time.

Heat flow: differential scanning calorimetry (DSC) test

Different crucibles were used for sealers according to whether moisture was required for setting. Freshly mixed sealer was dispensed into a preweighed empty aluminum crucible (AH Plus, RoekoSeal, GuttaFlow2) or a prepared crucible lined with gypsum (Bioseal, iRoot SP). Each crucible filled with sealer was weighed again, fitted with a lid and then immediately placed in a DSC apparatus (Mettler-Toledo, Columbus, OH, USA) to undergo an isothermal scan at 37 °C for 24 h. The heat flow was automatically recorded every 2 s. An empty 40-μL crucible was used as a reference during measurement.

Statistical analysis

SPSS version 21.0 (SPSS Inc., Chicago, IL, USA) was used to analyze the data of film thickness, flow, working and setting time. Statistical analysis was performed using one-way ANOVA test. The level of significance was set at $P < 0.05$. The resulting thermogram was evaluated by the software from DSC manufacturer (Mettler-Toledo).

Results

Film thickness and flow

Bioseal showed the highest film thickness of 44 μm ($P < 0.05$), which was almost double to triple that of the other 4 tested sealers. Whereas the lowest film thickness of 13 μm was observed in iRoot SP ($P < 0.05$) (Table 2).

Bioseal and GuttaFlow2 had the lowest flow of 21.43 and 22.31 mm, respectively ($P < 0.05$). While the highest flow was detected in iRoot SP and RoekoSeal, with values of 27.35 and 27.20 mm ($P < 0.05$) (Table 2).

Working and setting time

For each tested sealer, the working time at 37 °C was shorter than that at 23 °C ($P < 0.05$) (Table 3). The working time of Bioseal was 8.2 min at 23 °C and 4.5 min at 37 °C ($P < 0.05$) (Fig. 1).

Regarding setting time, Bioseal had the shortest value among the tested sealers of 16.3 min ($P < 0.05$), whereas GuttaFlow2 and RoekoSeal exhibited results of 23.2 and 46.5 min respectively. Longer setting time of 398.3 and 571.7 min were observed in AH Plus and iRoot SP ($P < 0.05$) (Fig. 1).

Heat flow

The results of differential scanning calorimetry (DSC) test are illustrated in the thermogram (Fig. 2). It demonstrated that the setting process of all the 5 tested sealers was exothermic. Bioseal had the greatest heat intensity of 10.5 mW/g, almost 1.5–2.5 times as much as that of the other 4 tested sealers ($P < 0.05$). Bioseal reached the heat flow peak at 14 min and ended at nearly 25 min, followed by GuttaFlow2 and RoekoSeal, whose exothermic peak appeared at 30 and 44 min respectively. Whereas AH Plus and iRoot SP showed exothermic peak at 4.3 and 5.6 h, and their heat flow ended at approximately 11 and 16 h respectively.

Table 3 Working time of the 5 tested sealers (mean ± standard deviation).

	Working time (min)	
	23 °C (50% humidity)	37 °C (95% humidity)
Bioseal	8.2 ± 0.3[A d]	4.5 ± 0.5[B d]
iRoot SP	164.3 ± 4.0[A a]	105.0 ± 3.6[B a]
AH Plus	123.7 ± 3.2[A b]	82.3 ± 4.2[B b]
RoekoSeal	22.2 ± 0.3[A c]	14.2 ± 0.8[B c]
GuttaFlow2	11.3 ± 0.6[A d]	6.8 ± 0.3[B d]

[A,B] Different letters in the same row and [a,b,c,d] different letters in the same column indicate statistically significant differences ($P < 0.05$).

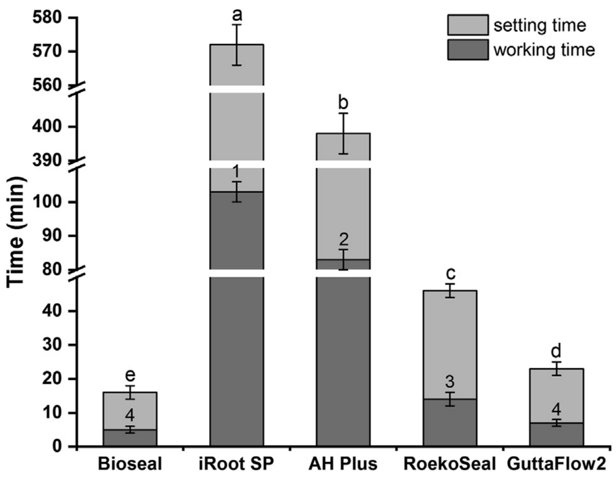

Figure 1 Working and setting time of 5 root canal sealers (at 37 °C). ^{a,b,c,d,e} Different letters and$^{1-4}$ numbers of the same-colored column showed statistically significant differences ($P < 0.05$).

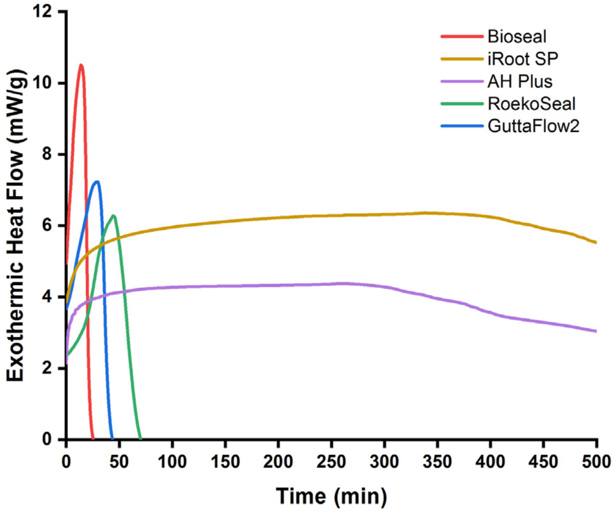

Figure 2 Graphical representation of the heat flow during setting process of 5 root canal sealers.

Discussion

With novel root canal sealers being developed, it is important for clinicians to understand their physicochemical properties. The sealer Bioseal was developed on the basis of silicone-based sealer RoekoSeal and flowable gutta-percha system GuttaFlow2, which has gutta-percha powder that is less than 30 μm in diameter. Strictly speaking, Bioseal is a multicomponent sealer that contains bioceramic particles $CaO-SiO_2-Na_2O-ZrO_2-P_2O_5$ in the range of 20—40 μm. In this study, physicochemical properties, including film thickness, flow, working and setting time, as well as thermal characteristic in the setting process of Bioseal were evaluated. The characteristics of Bioseal were also compared with the commonly used epoxy resin-based sealer AH Plus and the bioceramic-based sealer iRoot SP.

Film thickness and flow of a sealer represent its ability to enter narrow and irregular spaces.[1] A sealer with large film thickness and low flow may have difficulty filling anatomical irregularities, the isthmus and lateral canals.[9] While a sealer with the opposite characteristics may result in sealer extrusion over the apical foramen.[10,11] Both film thickness and flow are affected by factors such as constituent compositions, particle size and ambient temperature.[12] Among the 5 tested sealers in the present study, bioceramic silicone-based sealer Bioseal showed the highest film thickness of 44 μm and the lowest flow of 21.43 mm. This result might be attributed to the ingredient components and particle size. Bioseal contains bioceramic particles with a diameter of 20—40 μm and gutta-percha powder in diameter of 30 μm.[13] Meanwhile, bioceramic-based sealer iRoot SP, whose particle size is 2 μm,[14] had excellent flow of 27.35 mm and the lowest film thickness of 13 μm among the 5 tested sealers. It has been confirmed that iRoot SP could penetrate into the dentinal tubules,[15] which have diameters ranging from 1 to 3 μm.[16] The effect of the characteristic film thickness and flow of Bioseal on its clinical performance is still uncertain and warrants further investigation.

According to the ISO 6876:2012 specifications, working time is defined as the length of time from sealer mixing until the ductility decreases to 17 mm and the sealer becomes unworkable at 23 °C. Considering that sealers commonly used in practice are injectable and ready-to-use, working time was measured at both 23 °C and 37 °C in the present study. The results showed that working time at 37 °C was shorter than that at 23 °C for each sealer. This finding is in accordance with the principle of thermodynamics that an increase in temperature accelerates chemical reactions.[17—19]

At 37 °C, Bioseal had the shortest working time of 4.5 min, which may put pressure on practitioners. As reported that the mean obturation time by senior dentists was 3.5 min for lateral compaction and 2.5 min for continuous wave of obturation in single-rooted teeth.[20,21] While for dental students, the average time for lateral compaction and single-cone obturation in maxillary incisors was approximately 8 and 6 min, respectively.[22] Therefore, clinicians should consider canal anatomy, obturation techniques and practitioner experience when using sealer Bioseal, which has short working time.

In this study, differential scanning calorimetry (DSC) test was used to measure the heat flow during setting reaction of sealers. DSC is a thermal analysis technique well suited for the study of chemical reactions and setting time in a wide range of materials. In the present study, Bioseal was observed to have the greatest and earliest exothermic peak among the 5 tested sealers. In the thermogram, the time when the peak was reached indicated most chemical reaction completion and the main product formation.[23] At this time, physicochemical properties of the sealer tended to stabilize, and it has been defined as initial setting in clinical practice.[24] Interestingly, the results showed that Bioseal reached the heat flow peak at 14 min, in accordance with its setting time of 16.3 min measured by ISO specifications. Furthermore, the heat generation peak in Bioseal was almost 1.5—2.5 times as much as the exothermic reaction of the other 4 kinds of sealers. The reason for this finding might be that Bioseal is a multicomponent sealer that contains bioceramics and silicone. Therefore, it is set by both hydration reaction and polymerization reaction, which may lead to an increase in temperature. In turn, this increase in temperature accelerates the setting process, releases more heat in a short time and becomes a closed loop. This may partly explain the strong exotherm and short setting time observed for Bioseal. In addition, the time for post space preparation after root canal obturation is determined by the final setting of sealers, in terms of the time when the heat flow shown in the thermogram ends completely.[25] The heat flow of Bioseal lasted nearly 25 min, while for AH Plus and iRoot SP, their setting reaction ended at 11 and 16 h, respectively. Different setting time provides useful information for clinicians when choosing material for immediate tooth intracanal restoration after root canal obturation.

In conclusion, novel bioceramic silicone-based sealer Bioseal showed large film thickness, low flow, short working and setting time, with intense and fast exothermic setting reactions. Clinicians should take it into consideration when using sealer Bioseal in daily work.

Declaration of competing interest

The authors have no conflicts of interest relevant to this article.

Acknowledgments

The work was supported by Department of Cariology and Endodontology, Peking University School and Hospital of Stomatology, China.

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