Effects of heating on the physical properties of premixed calcium silicate-based root canal sealers

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Introduction

The purpose of root canal filling is to tightly seal a root canal with a biocompatible material after eliminating the source of infection, thereby entombing the remaining bacteria in the root canal and preventing ingress of microorganisms from the oral environment and their passage to periapical tissues [1]. Root canal sealers are essential for tightly sealing the gap between the root canal wall and solid root canal filling materials such as gutta-percha [2]. Because long-term tight sealing of the root canal depends on the properties of the sealer [3], the materials used in the sealing procedure need to be physically stable.

Adaptation of gutta-percha to the root canal wall may be better with the warm vertical compaction technique than with the lateral condensation and single-cone techniques, as it allows for a homogeneous and dense root canal filling that adapts to the irregularities of root canals [4-7]. However, heat has been reported to alter the physical properties of some root canal sealers and the composition of the set products [8-14]. In particular, data for the epoxy resin root canal sealer AH Plus (DeTrey Dentsply, Konstanz, Germany) indicate shortened setting time and increased film thickness [9-11,14], as well as chemical changes such as loss of the amino group and changes in the structure of the resin [9,10,11,13].

Thus far, various types of root canal sealers have been used extensively, including those based on zine oxide eugenol, resin, glass ionomers, silicate, and calcium hydroxide. In recent years, however, root canal sealers mainly composed of calcium silicate have been attracting much attention in clinical practice. Calcium silicate-based root canal sealers are generally considered to have beneficial properties, such as biocompatibility, low toxicity, high alkalinity, and resistance to shrinkage [15-18]. In addition, because premixed calcium silicate-based root canal sealers do not need to be mixed, a homogeneous root canal sealer can be administered directly from the syringe into the root canal. Premixed sealers are primarily intended to be used in single-cone technique; however, the recently developed EndoSequence BC Sealer HiFlow (Brasseler, Savannah, GA, USA; BC Sealer HiFlow) is considered suitable for the warm vertical condensation technique. A previous report showed that when BC Sealer HiFlow was heated, flow was higher than that of a preceding product (EndoSequence BC Sealer, Brasseler; BC Sealer), and film thickness was lower [18].

However, little is known about heat-induced changes in the physical properties of calcium silicate-based root canal sealers. This study therefore examined the effects of heat on the physical properties of four types of calcium silicate-based root canal sealers with different compositions, which were used as test materials in the study. The null hypothesis was that heating does not change the physical properties of calcium silicate-based root canal sealers.

Materials and Methods

Four types of calcium silicate-based root canal sealers—Endoseal MTA (Maruchi, Wonju, Republic of Korea), Well-Root ST (Vericom, Gangwon-Do, Republic of Korea), EndoSequence BC Sealer (BC Sealer), EndoSequence BC Sealer HiFlow (BC Sealer HiFlow)—and an epoxy resin root canal sealer, AH Plus (DeTrey Dentsply, Konstanz, Germany), were tested. The compositions of the sealers are shown in Table 1. Setting time, flow, and film thickness were examined according to ISO 6876:2012 [19] at different temperatures. Each experiment was performed eight times.

Setting time

AH Plus does not require moisture for setting and was therefore mixed and inserted into a stainless steel mold (inner diameter: 10 mm, height: 2 mm) [17,20]. In contrast, BC Sealer, BC Sealer HiFlow, Well-Root ST, and Endoseal MTA require moisture for setting; therefore, in accordance with ISO 6876:2012 [19], they were dispensed into gypsum molds (inner diameter: 10 mm, height: 1 mm). Measurement of the setting started after the mold was filled up, and samples were stored and classified in relation to whether they were placed in a 37°C incubator (TVN480DA, Advan-tec, Tokyo, Japan) or an oven (007S, KDF, Kyoto, Japan) at 100°C for 1 min and later transferred into an incubator at 37°C and 95% humidity. The molds designed for the tests conducted at 100°C were heated in the oven before use. Setting time was measured by using the method specified in ISO 6876:2012, i.e. a Gilmore-type metric indenter (weight: 100 ± 0.5 g; tip diameter: 2 ± 0.1 mm) was slowly lowered onto the surface of the samples at 1-min intervals to determine the time point when the indenter failed to make a visible indentation.

Flow

A syringe was used to place the sealer (0.05 ± 0.005 mL) on the center of a glass plate (dimensions: 40 mm × 40 mm, thickness: 5 mm), and 180 ± 5 s later, the sealer was overlaid with a similar glass plate. The total weight was adjusted to 120 ± 2 g by adding weight (100 g). Ten minutes after placement of the sealer had begun, the added weight was removed, and the compressed sealers maximum diameter and minimum diameter were...
measured with ImageJ software (Ver 1.52s, National Institutes of Health, Bethesda, MD, USA). Each sealer was measured at room temperature (23 ± 2°C), and pH was measured with a pH reference value and were significantly higher than the values for the other groups (P < 0.05). Data processing was performed by using SPSS version 22 (SPSS Inc., Chicago, IL, USA). Normality was tested with the Kolmogorov-Smirnov test, and equality of variances was tested with the Levene test. One-way ANOVA, followed by the Tukey test, was used to compare the physical properties of sealers. The independent t-test was used to compare differences between two conditions. To analyze pH values, the Kruskal-Wallis test followed by the Mann-Whitney U-test with Bonferroni correction was used. A P-value of <0.05 was considered to indicate statistical significance.

**Results**

Table 2 shows the means and standard deviations for setting time, flow, and film thickness in environments at different temperature levels.

**Flow**

According to ISO 6876:2012, the requirement for flow is at least 17 mm. In the present experiments, all sealers met this standard. AH Plus had the longest setting time (730 ± 9.2 min), and Endoseal MTA had the shortest setting time (2.5 ± 0.3 min). When placed in a 100°C environment for an initial 1 min, all sealers exhibited significantly shortened setting times (P < 0.05). Furthermore, while BC Sealer (55 ± 5.5 min) and BC Sealer HiFlow (68 ± 15 min) attained the standard value, the setting times for Endoseal MTA (2.5 ± 0.3 min) and Well-Root ST (14 ± 2.9 min) were below the recommended value.

**Film thickness**

According to ISO 6876:2012, the requirement for film thickness is 50 μm or lower. In the present experiments, all sealers attained this reference value at room temperature. However, when the sealers were placed at 100°C for an initial 1 min, Endoseal MTA (308.9 ± 107.1 μm) and Well-Root ST (63 ± 18.7 μm) had film thickness values that both exceeded the reference value and were significantly higher than the values for the other groups (P < 0.05).

**Microstructure and elemental composition of the set sealers**

Figures 1 and 2 show the results of SEM and EDS analyses of sealers allowed to set at different temperatures. In all sealers, the film thickness was below the standard value for all sealers, and was significantly reduced (P < 0.05). Flow was completely lost in Endoseal MTA, and the measurements could not be performed. The flow rates of Well-Root ST (16.7 ± 1.1 mm) and BC Sealer HiFlow (12.3 ± 1.4 mm) were significantly higher than those observed in other groups (P < 0.05).
ferences did not cause apparent changes in surface texture or elemental concentrations.

SEM images of AH Plus showed that it was composed of a matrix and numerous, relatively large, white particle images. EDS analysis identified carbon, oxygen, calcium, zirconium, and tungsten as the main constituent elements. The white particles contained large amounts of calcium and tungsten.

SEM images of Endoseal MTA showed a mixture of particles of various morphologies. EDS analysis identified the presence of carbon, oxygen, magnesium, aluminum, silicon, calcium, zirconium, and bismuth. The white particle images contained a large amount of bismuth.

SEM images of Well-Root ST showed many white particles scattered in the matrix, and EDS analysis revealed they were composed of carbon, oxygen, aluminum, silicon, calcium, titanium, and zirconium. The white particle images contained a large amount of zirconium.

SEM images of BC Sealer and BC Sealer HiFlow were similar and exhibited homogeneous particle aggregates containing a few white particle images. EDS analysis showed that both sealers were composed of carbon, oxygen, silicon, calcium, and zirconium.

**pH measurement**

Table 3 shows mean pH values and standard deviations recorded during water immersion of sealers set in different temperature environments. The pH values of calcium silicate-based root canal sealers were higher, with some significant differences ($P < 0.05$), than those of AH Plus, while no significant difference was found among calcium silicate-based root canal sealers ($P > 0.05$). The results of tests of all sealers showed no significant difference between the pH of the heated and non-heated groups ($P > 0.05$).

**Discussion**

This study demonstrated that when the sealers were heated, significant changes in setting time, flow, and film thickness were observed in all the sealers tested. Therefore, the null hypothesis was rejected.
Heating the plugger to approximately 200°C is recommended when performing warm vertical condensation technique [21]. However, previous studies reported that when the System B plugger was used at a temperature setting of 200°C, the actual temperature was approximately 100°C and did not reach 200°C [8,9]. In addition, the mean temperature in various types of heat pluggers was reported to be approximately 140°C [20]. In the present study, the environmental conditions was simulated by using conditions that involved heating at 100°C for 1 min [10], as well as preheating glass plates and molds at 100°C to perform direct heating.

In this study, setting time was significantly reduced in all sealers when heating was applied. Similar to the previous findings, a previous study reported that the setting time for AH Plus was shortened when heating was applied [9-11]; this is attributable to heat-induced acceleration of the polymerization reaction of the resin. Meanwhile, setting time was shortened for calcium silicate-based root canal sealers subjected to heating, which was consistent with previous findings for iROOT SP (an equivalent of BC Sealer) [10]. Elevated temperatures promote hydration reactions in Portland cement [22], and a similar setting acceleration is assumed to occur in calcium silicate-based root canal sealers.

ISO 6876: 2012 standards [19] recommend a setting time of at least 30 min, to allow enough time for a root canal filling procedure. However, the setting time of Endoseal MTA was reduced to approximately 2.5 min when it was subjected to heating. The setting mechanisms of Endoseal MTA include not only the hydration reaction of calcium silicate but also the pozzolanic reaction (conversion of calcium hydroxide to insoluble phases such as calcium silicate hydrate) [23], which is believed to allow for rapid setting [24]. However, the present results suggest that use of this product for warm vertical compaction technique may not allow sufficient time to complete the procedure. In addition, Well-Root ST exhibited rapid setting, second only to the setting rate of Endoseal MTA. This is attributable to the fact that Well-Root ST contains calcium alumino-silicate, which is capable of setting more rapidly than calcium silicate. In contrast, BC Sealer contains calcium dihydrogen phosphate and calcium hydroxide, in addition to calcium silicate. Thus, hydroxyapatite and water are produced after the reaction between calcium dihydrogen phosphate and calcium hydroxide during setting, and the water generated, in addition to the moisture in the environment, accelerates the hydration reaction of calcium silicate [13]. The setting reaction of BC Sealer has not been reported; however, according to the manufacturer, it is similar to that of BC Sealer (https://media.brasselerusa.com/userfiles/1F%20Manuals%2CBrochures/SDS-00001%20EndoSequence%20BC%20Sealer%20SDS%20%20REV%20%20G.PDF).

The present EDS analysis confirmed that BC Sealer and BC Sealer HiFlow had similar elemental compositions. However, a recent study reported that heating at 100°C for 1 min did not affect setting times for BC Sealer and BC Sealer HiFlow [18], which is inconsistent with the present results. This discrepancy can be explained by the fact that, in this study, heating had a clear impact because the mold was preheated to 100°C before conducting the experiments.

The flow of root canal sealers is believed to be dependent on particle size, shear rate, temperature, time, the inner diameter of the root canal, and the insertion speed [1]. An adequate flow during the time allocated to the procedure is essential to seal irregularities in the root canal, as well as the apical area [1]. The present flow values at room temperature were comparable to those of previous reports [25,26] and met the ISO 6876:2012 criterion [19] of >17 mm. In contrast, heating caused a significant decrease in flow in all sealers, because the setting reaction is accelerated at elevated temperatures [10,22]. The flow of all sealers subjected to heating in the present study did not meet the aforementioned ISO 6876:2012 criterion [19]. Flow could not be measured for Endoseal MTA, which suggests that this product should not be used in warm vertical condensation technique. When BC Sealer HiFlow was subjected to heating, flow was significantly higher than in BC Sealer, which was consistent with previous findings [18]. When Endoseal MTA and Well-Root ST were heated, film thickness exceeded 50 μm. This increased film thickness after heating is explained by heat-induced acceleration of setting time. Because sealers tend to dissolve over time, use of the minimum amount is advised, and thin sealers are preferred [27]. In contrast, calcium silicate cements tend to expand by approximately 0.2% to 6% [28]. Further, the sealing ability of a sealer in relation to its thickness was reported to vary by sealer type [29]. In addition, calcium silicate-based root canal sealers have been reported to be more soluble than AH Plus and to release greater amounts of calcium ions [30]. The reaction between released calcium ions and surrounding phosphate ions generates apatites, which deposit as a precipitate on the surface layer of the sealer [31,32]. This is assumed to contribute to the high biocompatibility of calcium silicate-based root canal sealers [16,18]. However, dissolution of a thick layer of sealer might be unfavorable for long-term maintenance of root canal sealing ability.

SEM images and EDS analyses showed no apparent heat-induced compositional alterations or ultrastructural changes. Although differences between various types of sealers with respect to particle size, composition, and surface texture were observed, BC Sealer and BC Sealer HiFlow did not clearly differ.

As is the case for MTA, hydration of calcium silicates is the main reaction responsible for setting calcium silicate-based root canal sealers. When the generated calcium hydroxide comes into contact with water or tissue fluid, hydroxide ions are released along with calcium ions, leading to an alkaline pH [16,17,32-34]. When calcium silicate-based root canal sealers are subjected to heating, the hydration reaction is accelerated. Thus, the amount of calcium hydroxide produced at the initial stage of setting increases, which might affect the number of ions released. However, under the present experimental conditions, there was no impact on pH. An alkaline pH may contribute to antimicrobial properties [35] and induction of calcification [36], but the present findings suggest that heating has no effect on these properties.

Under the present experimental conditions, heating calcium silicate-based root canal sealers at 100°C for 1 min accelerated setting time, decreased flow, and increased film thickness; however, the extent of these changes varied among the products. These changes in physical properties could adversely affect the quality of root canal filling when calcium silicate-based root canal sealers are used for warm vertical condensation technique.

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Conflict of interest
The authors declare no conflict of interest.

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