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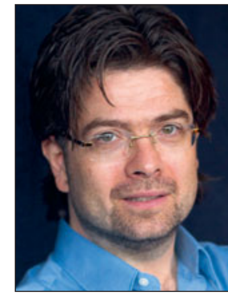
Evaluating the physical properties of one novel and two well-established epoxy resin-based root canal sealers



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Objectives: Despite the variety of root canal sealers on the market, an ideal root canal sealer has not been developed. A new epoxy resin sealer, BJM Root Canal Sealer, contains a quaternary amine macromolecule, Biosafe, which provides anti-biofilm features. The aim of the present study was to perform an *in vitro* evaluation and comparison of the physical properties of this novel endodontic epoxy resin-based sealer (BJM Root Canal Sealer) with those of AH Plus and MM-Seal.
Materials and methods: Flow, working time and solubility were evaluated based on the ISO 6876/2012 standards. Dimensional change was assessed based on the ISO 6876/2001 standards. The physical properties were analysed using one-way analysis of variance (ANOVA).
Results: The tested sealer flows were consistent with the ISO 6876/2012 recommendations. The dimensional changes of the tested sealers were inconsistent with the ISO 6876/2001 recommendations. The AH Plus sealer exhibited a lower flow than the other sealers ($P < 0.05$). The MM-Seal sample featured the lowest working time and highest dimensional change compared with the other sealers ($P < 0.05$). Solubility tests showed that AH Plus and BJM Root Canal Sealer have solubilities within the limit allowed by the ISO 6876/2012 recommendations (3% mass fraction).
Conclusions: BJM Root Canal Sealer presented physicochemical properties that were similar to the AH Plus sealer. The physicochemical properties of BJM Root Canal Sealer conformed to the ISO 6876/2012 recommendations, except for the dimensional change, which did not fulfil the ISO 6876/2001 recommendations, as seen in all tested sealers.

Introduction

The goals of obturation are to fill the root canal system with an impervious, biocompatible and dimensionally stable seal[1]. A root canal sealer is an important component in root canal obturation. Ideally, a root canal sealer should produce a three-dimensional obturation throughout the canal by providing adherence between the gutta-percha and root canal walls, which prevents gaps at the sealer/dentine interface and, therefore, leakage[2-6]. Currently, various types of endodontic sealers are available, including sealers based on zinc oxide – such as zinc oxide eugenol (ZOE) – resin, glass ionomer, calcium hydroxide, silicone, mineral trioxide aggregate (MTA)-based, and bio-ceramic root canal sealers.

Despite the variety, the ideal root canal sealer has not yet been discovered. According to Grossman, an ideal root canal sealer should provide an excellent seal when set, dimensional stability, a slow setting time to ensure a sufficient working time, insolubility to tissue fluids, adequate adhesion with canal walls, and biocompatibility[7]. An additional desirable property is antibacterial activity, which may provide an additional reduction in bacteria and thus improve success[8-10].

Epoxy resin sealers have been extensively evaluated for their physicochemical properties and

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biological response. The current modifications to the original formula are widely used for root canal filling procedures[11-15].

Recently, a quaternary ammonium macromolecule (Biosafe HM4100, Biosafe, Pittsburg, PA, USA) was incorporated into a novel epoxy resin-based sealer, BJM Root Canal Sealer (BJM Laboratories, Or-Yehuda, Israel). Biosafe provides long-lasting surface protection against bacteria, fungi, and viruses[16]. Further studies about Biosafe have not yet been published. In this study, the physical properties of three epoxy resin root-canal sealers were compared: BJM Root Canal Sealer; the MM-Seal sealer (Micro Méga, Besançon, France); and AH Plus sealer (Dentsply DeTrey, Konstanz, Germany). The aim was to compare the physical properties of the BJM Root Canal Sealer with those of the AH Plus and MM-Seal sealers, based on the ISO 6876/2012 recommendations.

Materials and methods

Three types of epoxy resin root canal sealers were used as the experimental materials: AH Plus sealer, MM-Seal sealer and BJM sealer. The physical properties included flow, working time, solubility and dimensional change. Flow, working time and solubility were examined based on the ISO 6876/2012 specifications[17]. All sealers were mixed and manipulated in accordance with the manufacturers' instructions.

Flow

A mixed sealer with a volume of 0.05 ± 0.005 mL was placed in the centre of a glass plate (40 × 40 × 5 mm^3) using a graduated disposable 3 mL syringe. After 3 min, a second glass plate weighing 20 g and a 100 g weight were placed centrally on top of the sealer. Ten minutes after mixing began, the load was removed and the sample disk's minimum and maximum diameters were measured using a digital micrometre (Swiss Precision Instruments, Los Angeles, CA, USA) with a 0.01 mm resolution. If the diameters were within 1.0 mm of each other, the mean of the two diameters was recorded. If the two diameters were not within 1.0 mm of each other, the test was repeated. The flow of the sealer was determined based on the mean of five tests for each sealer.

Working time

The working time test was performed using the same procedure used for flow, with the exception that the load was applied 15 s before the end of the manufacturer's stated working time. For each experimental sealer, five samples were constructed, and the mean was considered the sample working time.

Solubility

The sealer solubilities were investigated as a percentage of the specimen material mass removed from distilled water compared with the original mass of the specimen.

Two split ring moulds with an internal diameter of 20.0 mm and a height of 1.5 mm were made of stainless steel placed on a glass plate. The rings were filled with the sealers, and another glass plate faced with a plastic sheet was placed on top of the sealer and carefully removed to leave a flat, uniform surface. The filled moulds were placed in a cabinet maintained at 37°C and 95% relative humidity (RH) for a period of time that was 50% longer than the setting time stated by the manufacturer. The specimens were removed from the mould and the mass of sealer was determined to the nearest 0.001 g. Two specimens were placed in a petri dish and 50 mL of water was added and the dish was covered. The dish was placed in the cabinet for 24 h. A fluted filter was placed into a funnel and the funnel was placed 20.0 mm above the bottom of the second petri dish. The water was poured together with the specimens into the fluted filter. The previously used dish was washed three times with 5 mL of water and the water was poured into the fluted filter.

Along with the collected water, the second petri dish was placed, in an oven at 110°C, the water was evaporated to constant mass, and the dish was cooled in the desiccator to room temperature before each weighing.

The difference between the original mass of the shallow dish and its final mass, to the nearest 0.001 g, was recorded as the amount of sealer removed from the specimens. The difference in mass was recorded, calculated as a percentage of the original combined mass of the two specimens, to the nearest 0.1%, and the mean value indicated the solubility.

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Table 1 Sealer physical properties (mean ± standard deviation).

	AH Plus	MM-Seal	BJM Root Canal Sealer
Flow (mm)	20.18 ± 0.72[a]	22.71 ± 1.01[b]	23.22 ± 0.45[b]
Working Time (min)	18.32 ± 2.12[a]	19.45 ± 2.53[a]	20.12 ± 2.13[a]
Solubility (%)	-2.49 ± 3.30[a]	-3.85 ± 3.80[a]	1.31 ± 0.50[b]
Dimensional Change (%)	-2.12 ± 0.91[a]	-12.2 ± 3.03[b]	-5 ± 2.34[a]

Different superscript letters in each test indicate a significant difference ($P < 0.05$).

Dimensional change

Dimensional change was evaluated according to ISO 6876/2001 recommendations because ISO 6876/2012 recommendations do not include dimensional change. Dimensional change was measured for over 30 days using cylindrical specimens that were constructed in split moulds that were 6.0 mm in diameter and 12.0 mm high. The moulds were slightly overfilled with mixed sealer and backed by a thin polyethylene plastic sheet, as well as a glass plate on each side. The moulds and plates were held firmly together with a clamp. Five minutes after beginning the mix, the mould with the sealer and clamp was transferred to an incubator (37°C, > 95% RH) and maintained in the incubator. After setting, the ends of the moulds containing the specimens were ground with 600-grit wet sandpaper. The specimens were then removed from the mould, and the length was measured again with a digital micrometre (Swiss Precision Instruments). The specimens were stored separately in polyethylene tubes that contained 50 mL of distilled water and were maintained in an incubator (37°C, > 95% RH) for 30 days. The samples were then removed from the distilled water, blotted dry and measured again for length. The results from the five samples were averaged.

Statistical analysis

The physical properties were analysed via a one-way analysis of variance (ANOVA) with SPSS software 17.0 (SPSS, Chicago, IL, USA). The significance level was $P < 0.05$.

Results

The physical properties of the sealers are summarised in Table 1. The flow test showed that the tested sealers conformed to the ISO 6876/2012 standards, which states that the sealer should have a diameter of no less than 17.0 mm. Statistical analyses showed that BJM Root Canal Sealer exhibited a higher flow (23.22 ± 0.45 mm) than MM-Seal ($P < 0.05$), and AH Plus exhibited a significantly lower flow (20.18 ± 0.72 mm) than the other sealers ($P < 0.05$). In increasing order the flow values were: AH Plus < MM-Seal < BJM Root Canal Sealer.

Working time was determined by performing the flow test 15 s before the end of the stated working time. The diameter in the flow test should be no less than 17.0 mm, 15 s before the end of the stated working time. According to the manufacturers, AH Plus and BJM Root Canal Sealer sealers produced the same working time values, while the MM-Seal exhibited a lower working time (35 min). The working time for all the sealers was greater than 17.0 mm 15 s before the end of the stated working time. The working time values, in increasing order, were MM-Seal < BJM RCS < AH Plus.

Solubility tests showed that AH Plus and BJM RCS sealers exhibited solubilities within the limit allowed by the ISO 6876/2012 recommendations (3% mass fraction). The solubility values, in increasing order, were MM-Seal < AH Plus < BJM Root Canal Sealer.

Based on the dimensional change test, neither sealer conformed to the ISO 6876/2001 recommendations, wherein the mean linear shrinkage of the sealer should not exceed 1% or 0.1% in expansion. AH Plus and BJM Root Canal Sealer produced the same dimensional change values, while MM-Seal generated a significantly higher dimensional change value (-12.2% ± 3.03%) than the other

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sealers ($P < 0.05$). The dimensional change values, in increasing order, were AH Plus < BJM Root Canal Sealer< MM-Seal.

Discussion

The goal of root canal treatment is to cure or prevent periapical disease[18]. Success may be defined as a significant microbial reduction in the infected root canals[19-21]. Methods currently available for reducing the bacterial population within the root canal system include a combination of mechanical instruments, irrigation, agitating irrigation with various solutions, and the application of antibacterial medication in the root canal, but full eradication remains unattainable[22-24].

Antibacterial activity is a desirable property of root canal filling materials that may provide an additional decrease in bacteria and improve success[8-10]. The antibacterial properties of root canal sealers have been widely studied[25-26].

Epoxy resin sealers exhibit antibacterial activity during setting; while sealers based on calcium hydroxide or ZOE exhibit a longer antibacterial influence due to their active ingredients. However, these sealers tend to dissolve over time and thus may compromise the seal[27]. Recently, nanoparticles were successfully added to AH Plus for anti-biofilm features[28-30].

Kenawy et al introduced a new approach to insoluble macromolecular disinfectants, which can inactivate target microorganisms through direct contact, without releasing antibacterial agents. The mechanism is based on the positive charge of the macromolecules, which interacts with, and thus disrupts, the negatively charged bacterial membrane[31]. One of the macromolecules most used in general medicine materials is Biosafe, which is widely used as an additive to catheter and keypad cover plastic materials to prevent biofilm formation[16]. The Biosafe macromolecule was incorporated into the novel epoxy resin-based BJM Root Canal Sealer to impart additional antibacterial features. The characteristics and physicochemical properties of sealers are fundamental to their clinical behaviour. Several standardized tests must be performed to examine their physical and biological properties before clinical use[1]. For flow, all tested sealers exhibited acceptable values based on the ISO 6786/2012 recommendations[17], in which the minimal flow required for sealer is 17.0 mm.

Flow is an important physical property that indicates the capacity of the sealer to enter and fill spaces that are difficult to access, such as the isthmus and accessory canals. However, a high flow might increase the potential that the material will be extruded toward the periapical region[32].

In the present study, all tested sealers conformed to the ISO 6876/2012 standards, wherein the sealer should have a diameter of no less than 17 mm. AH Plus exhibited a significantly lower flow (20.18 ± 0.72 mm) than the other sealers. The flow of AH Plus in the present study was similar to one investigated in a previous study[33]. According to ISO 6876:2012[17], the working time is the period of time measured from when mixing begins and during which the dental sealer can be manipulated without an adverse effect on its properties. This time is directly linked to the setting time, which is primarily a control test for the stable behaviour of a product, and depends on the constituent components, their particle size, the ambient temperature and RH[34]. In the present study, all sealers conformed to the ISO 6876/2012 standards – the working time for all the sealers was greater than 17.0 mm 15 s before the end of the stated working time, similar to a previous study[33].

Solubility and dimensional changes are two important physical properties for the evaluation of sealer stability. Solubility is the chemical substance property that defines the capacity of a solute to dissolve in a solid, liquid, or gaseous solvent to form a solution of the solute in the solvent. In other words, solubility means a loss of mass during immersion in water[35]. In the present study, all sealers except MM-Seal produced acceptable solubility values based on the ISO 6786/2012 recommendations[17], wherein the solubility of the set sealer should not exceed 3% mass fraction.

A previous study conducted according to the International Standards Organization 6876 standard reported solubility values of 0.32% for AH Plus and 0.38% for MM-Seal, respectively[36]. Moreover, in the present study, BJM Root Canal Sealer exhibited a gain in mass during immersion in water, which should be investigated in further studies. A possible reason may be that positively charged macromolecules are attracted to negatively charged OH- ions produced by dissociation of molecules in water.

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Dimensional change demonstrates material shrinkage or expansion following setting[1]. Dimensional change was not mentioned in the ISO 6786/2012 specification and, therefore, was evaluated according to the ISO 6876/2001 recommendations. Based on the dimensional change test in the present study, none of the sealers conformed to ISO 6876/2001 recommendations, which requires that the mean linear shrinkage of the sealer does not exceed 1% or 0.1% in expansion. The AH Plus sealer showed the lowest dimensional change (2.12% ± 0.91%), which contrasts with the MM-Seal, which exhibited the highest dimensional change (12.2% ± 3.03%). The present results are in opposite to a previous study[33]. Therefore, dimensional changes should be re-evaluated.

Based on the present study, the flow and working time of the BJM Root Canal Sealer were suitable and as good as AH Plus. Additional *in vitro*, *ex vivo*, and *in vivo* re search must be conducted to estimate the performance of the BJM Root Canal Sealer to evaluate its antibacterial effects and activity duration.

In conclusion, the results of this study show that the BJM Root Canal Sealer exhibited suitable physical properties based on the ISO 6786/2012 recommendations. All tested sealers conformed to all of the ISO 6876/2012 recommendations, except for the recommendation for dimensional change that did not conform to all of the ISO 6876/2001 recommendations.

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