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ORIGINAL ARTICLE

Solubility and apical sealing characteristics of a new calcium silicate-based root canal sealer in comparison to calcium hydroxide-, methacrylate resin- and epoxy resin-based sealers

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Abstract

Aim. To assess and compare the water sorption, solubility and apical sealing ability of iRoot SP and three other widely used root canal sealers. ***Materials and methods.*** Solubility was assessed by immersing standardized samples of calcium silicate- (iRoot SP), calcium hydroxide- (Sealapex), methacrylate resin- (EndoREZ) and epoxy resin- (AH Plus) based sealers in distilled water and measuring weight gain and weight loss at 6 h, 24 h and daily for 14 days. Roots of extracted mandibular premolars ($n = 80$) were prepared with 0.04-taper nickel-titanium rotary files to a final size 40. Roots were then randomly divided into four experimental groups ($n = 18$) and two control groups ($n = 4$), root canal sealers were applied and apical leakage was assessed using the fluid filtration method. Data was analyzed using Kruskal Wallis analysis of variance and Mann-Whitney U-tests, with the level of significance set at $p \leq 0.05$. ***Results.*** EndoREZ exhibited the highest water sorption, followed by iRoot SP, Sealapex and AH Plus. Sealapex exhibited significantly higher solubility than the other sealers, whereas no significant differences in solubility levels were observed between the other three sealers tested. AH Plus exhibited significantly lower microleakage than Sealapex and EndoREZ, whereas no difference in microleakage was found between AH Plus and iRoot SP. ***Conclusions.*** In view of the study findings, all tested sealers except Sealapex met the ANSI/ADA's requirements for solubility and no difference was found between AH Plus and iRoot SP in terms of apical sealing ability.

Key Words: *apical leakage, calcium silicate-based sealer, fluid filtration, iRoot SP, solubility*

Introduction

The final step of endodontic treatment entails filling of the root canal system in order to prevent leakage from the oral cavity and periradicular tissues into the root canal system and preserve the health of periapical tissues, thereby ensuring the success of root canal treatment [1]. The use of gutta-percha in combination with an insoluble endodontic sealer remains the most accepted approach to the final obturation of the root canal [1]. It is well established that selection of an appropriate sealer will influence the outcome of endodontic therapy [2]. In order to reduce the chance of endodontic treatment failure, sealers must demonstrate good sealing ability and insolubility or low solubility.

Different endodontic sealers have been introduced to the dental community in an attempt to improve apical seal and to provide insolubility. Epoxy resin-based sealers such as AH Plus have been widely used for many years and have exhibited lower solubility and disintegration [3] and better apical sealing performance [4] than methacrylate resin, calcium hydroxide and glass ionomer sealers. EndoREZ, a dual-cure methacrylate resin-based sealer, has been recommended for use in conjunction with a resin-coated solid material in order to form a monoblock. Thus, it creates a stronger and more leak resistant bond to the root canal wall [5]. Although various kinds of endodontic sealers have been proposed as innovative filling materials, the ideal root canal sealer has yet to be found. A new sealer recently introduced for the obturation of endodontically treated teeth, iRoot SP, has been described by its manufacturer as an insoluble, radiopaque, aluminum-free material composed mainly of calcium silicate; however, little information is available about the solubility and sealing properties of this new material. The aim of the present study was to assess and compare the water

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sorption, solubility and apical sealing ability of this new calcium silicate-based sealer with widely-used calcium hydroxide-, methacrylate resin- and epoxy resin-based sealers.

Materials and methods

Sealers

Four different types of endodontic sealers were evaluated in this study, as follows: Epoxy resin-based sealer AH Plus (Dentsply DeTrey GmbH, Konstanz, Germany); Polymethacrylate resin-based sealer EndoREZ (Ultradent, South Jordan, UT); Calcium hydroxide-based sealer Sealapex (SybronEndo Corporation, Orange, CA); and Calcium silicate-based sealer iRoot SP (Innovative BioCreamix Inc, Vancouver, Canada).

Solubility testing

Solubility tests were performed in line with ISO 4049E [6] methodology. Samples of each sealer tested were mixed and poured into a 1.6 mm thick plastic ring mold with an inner diameter of 20 mm ($n = 12$). An impermeable nylon thread was inserted into the material and the mold was sandwiched between two glass plates covered with cellophane film and pressed together manually to ensure that the sealer material was spread uniformly throughout the mold and that no air bubbles remained. Sealers were prepared according to manufacturer's instructions. AH Plus was mixed using the AH Plus Jet mixing system. Sealapex was mixed in a 1:1 ratio. For the iRoot SP group, pre-mixed ready-to-use cement was injected. EndoREZ was mixed by the auto-mix syringe and light cured for 2 min (Optilux 500, Demetron Research Corporation, Danbury, CT) at 600 mW/cm^2 to create an immediate coronal seal according to manufacturer's instruction. After the completion of filling, EndoREZ samples were immediately placed in a nitrogen chamber for 2 h to ensure that the methacrylate based-sealer had set without the presence of inhibiting oxygen and then was stored at 37°C and 95% humidity for 24 h to allow the sealer to set completely. All other specimens were stored in an incubator (37°C, 95% relative humidity) for a period corresponding to 3-times their recommended setting times (waiting times for each sealer are as follows; iRoot SP 4 × 3 h, Sealapex 2 × 3 h, and AH Plus 8 × 3 h). After setting, specimens were carefully removed from the molds using a scalpel and placed in a dehumidifier for 24 h to ensure dryness. Specimens were weighed 3-times using a precision scale with an accuracy of 0.00001 g (Mettler Toledo, Columbus, OH) and the mean reading (M1) was recorded. After weighing, specimens were individually transferred to tubes containing 20 mL of deionized distilled water, taking care to avoid any contact between the specimen and the inner surface of the tube, and stored in a 37°C incubator. Weight gain or loss of each disk was measured at 6 h, 24 h and daily for 14 days. Maximum wet mass (M2) for each sample was obtained by removing it from the water, blotting it dry with absorbent paper and weighing it after 1 min. Constant dry mass (M3) was obtained by placing each sample in a dehumidifier and weighing it after 24 h. Weight gain (water sorption), weight loss (solubility) and water uptake were calculated for each disk using the following equations:

$$\text{Water sorption}\,(\%) = \frac{(\text{M2} - \text{M1})}{\text{M1}} \cdot 100$$

$$\text{Solubility}\,(\%) = \frac{(\text{M1} - \text{M3})}{\text{M1}} \cdot 100$$

$$\text{Water uptake}\,(\%) = \frac{(\text{M2} - \text{M3})}{\text{M1}} \cdot 100$$

Apical microleakage testing

Eighty extracted human mandibular premolars were sectioned transversally below the cemento-enamel junction using a water-cooled precision saw (Isomet 2000 Precision Saw; Buehler, Lake Bluff, IL) to obtain root segments of 15 mm in length. For root canal preparation, Revo-S (MicroMega, Besançon, France) nickel-titanium rotary instruments were used in a crown-down manner at a speed of 350 rpm. The operating sequence was as follows:

(1) A 10, K-File was used to create a guiding path.
(2) A 6% taper, size 25 instrument (SC1) was used to 2/3 WL.
(3) A 4% taper, size 25 instrument (SC2) was used to full WL.
(4) A 6% taper, size 25 instrument (SU) was used to full WL.
(5) A 6% taper, size 30 instrument (AS30) was used to full WL.
(6) A 6% taper, size 35 instrument (AS35) was used to full WL.
(7) A 6% taper, size 40 instrument (AS40) was used to full WL.

Canals were irrigated with 3 mL of 5.25% NaOCl, followed by 5 mL 17% EDTA to remove the smear layer, between instrumentation and with 5 mL of distilled water to avoid the prolonged effect of the EDTA and NaOCl solutions after instrumentation was complete. Canals were dried with paper points. Roots were randomly divided into four experimental and two control groups, as follows:

(1) Gutta-percha with iRoot SP ($n = 18$).
(2) Gutta-percha with Sealapex ($n = 18$).
(3) Gutta-percha with AH Plus ($n = 18$).
(4) Resin-coated gutta-percha with EndoREZ ($n = 18$).

(5) Positive control (unfilled) ($n = 4$).
(6) Negative control (gutta-percha with AH Plus) ($n = 4$).

Each sealer was prepared and introduced into the canals according to the manufacturer's instructions. In all groups (except the positive control), pre-fitted size #40 0.06-tapered master cones (SybronEndo, Glendora, CA and Ultradent, South Jordan, UT) were laterally condensed with accessory cones. In the EndoREZ group, in line with the manufacturer's instructions, the coronal surfaces of the specimens were light-cured for 40 s to achieve an immediate coronal seal. Negative controls were filled with gutta-percha and AH Plus sealer. Positive controls were not obturated. For all experimental groups and positive controls, external root surfaces were coated with two layers of nail polish to within 2 mm of the apical foramen. For the negative control, the entire root surfaces, including apexes, were coated with three layers of nail polish. All specimens were stored at 37°C and 100% humidity for 1 week to allow sufficient time for the sealers to set.

Apical microleakage was assessed using an endodontic fluid transport model described by Wu et al. [7]. Each root was mounted by its apex to a plastic tube attached to a micropipette filled with deionized water. Cyanoacrylate glue was used to secure the root, tube and all connections of the testing apparatus. Compressed air was used to generate a constant pressure of 1 atm, a small air bubble was introduced into the system using an air syringe and positioned using a water syringe and the system was closed with hemostats. Fluid flow through the root fillings was measured by the movement of the bubble within a micropipette, which was recorded at 6, 8, 10 and 12 min. The average of the four measurements was calculated and recorded as microliters per minute (μL/min).

Statistical analysis

Shapiro-Wilk test showed an abnormal assumption of the solubility data; therefore, solubility data was analyzed using Kruskal-Wallis analysis of variance and Bonferroni's correction was used for post-hoc testing. Pair-wise multiple comparisons were performed using the Mann-Whitney U-test. Microleakage data was evaluated using Kruskal-Wallis analysis of variance and pair-wise multiple comparisons were performed using the Mann-Whitney U-test. All statistical analysis was performed using the software package SPSS 15.0 (SPSS Inc, Chicago, IL) (α set at 0.05).

Results

Water sorption and solubility

Water sorption, solubility and net water uptake rates of the endodontic sealers tested are given in Table I. EndoREZ exhibited the highest water sorption (4.18%), followed by iRoot SP (3.92%), Sealapex (1.70%) and AH Plus (0.30%). Differences in water sorption rates of sealers were significant ($p < 0.001$).

Table I. Water sorption and solubility of root canal sealers.

Materials		Water sorption % IQR	Solubility % IQR	Net water uptake % IQR
iRoot SP		3.92 (0.73)	0.90 (0.59)	4.78 (0.29)
Sealapex		1.70 (1.30)	4.44 (1.78)	6.09 (1.13)
EndoREZ		4.18 (1.10)	0.79 (0.45)	5.04 (1.37)
AH Plus		0.30 (1.41)	1.37 (1.46)	1.88 (1.07)
Comparisons between groups*	χ^2	32.892	26.176	30.774
	p	< 0.001	< 0.001	< 0.001

*Results of Kruskal-Wallis analysis of variance.

Solubility rates of the endodontic sealers tested are given in Table II. Sealapex (4.44%) showed significantly greater solubility than all other sealers tested ($p < 0.001$), exceeding the ADA specification of <3% solubility required for endodontic sealers. No significant differences were found between AH Plus (1.37%), iRoot SP (0.90%) and EndoREZ (0.79%). Net water uptake was the highest in Sealapex (6.09%) following EndoREZ (5.04%), iRoot SP (4.78%) and AH Plus (1.88%), respectively.

Apical microleakage

Microleakage data (μL/min) is given in Table III. Microleakage was lowest in the AH Plus group (0.35 ± 0.47) and highest in the Sealapex group (1.88 ± 0.69). With the exception of the microleakage levels of iRoot SP and AH Plus, highly significant differences ($p < 0.001$) in microleakage were observed between all groups (Table IV). Microleakage levels of controls confirmed the consistency of the experimental model, with no fluid transport observed in the negative controls and immeasurably rapid movement observed in the positive controls.

Discussion

The great variety of endodontic sealers that have been introduced to the dental community in an attempt to improve apical sealing and provide insolubility has

Table II. Post-hoc comparison of sealer solubility.

Materials	Sealapex Z (p)	EndoREZ Z (p)	AH Plus Z (p)
iRoot SP	3.974 (*<0.001*)	1.356 (0.190)	1.448 (0.151)
Sealapex		4.066 (*<0.001*)	3.694 (*<0.001*)
EndoREZ			1.850 (0.068)

p-values of different groups are shown in italics.

Table III. Apical leakage values of four root-canal sealants (μl/min).

Materials	Minimum (μl/min)	Maximum (μl/min)	Interquartile range (IQR)
iRoot SP	0.18	0.80	0.43 (0.38)
Sealapex	1.25	2.75	1.88 (0.69)
EndoREZ	0.93	1.88	1.21 (0.61)
AH Plus	0.10	0.93	0.35 (0.47)

made it necessary to provide assessments of the degrees of obturation and solubility of these materials [1,3]. Numerous solubility and leakage studies have been conducted for this purpose, but, surprisingly, very few studies have examined the solubility and sealing ability of iRoot SP. Furthermore, the advent of hydrophilic methacrylate resin-based root canal sealers and various hydrophilic MTA formulations has made water sorption of sealers an important issue. Ideally, endodontic sealers should exhibit low water uptake as well as low solubility; however, most studies examining the stability of endodontic sealers have looked only at solubility [3,8]. These studies measured the weight loss of test specimens in terms of the decrease in mass following storage in water [9], without taking into account that filler particles may be leached out of the specimens during storage in water and that water uptake may compensate for dissolved material [9,10]. For these reasons, the water sorption and solubility tests performed in the present study followed the procedures laid out in the ISO Standard 4049E [6], which is more appropriate than both the American Dental Association's ANSI/ADA Specification No.57 [11] and ISO Standard 6876 [12] for assessing methacrylate resin- and calcium silicate-based sealers. Furthermore, in line with Schafer and Zandbiglari's [3] observation that 24-h is an insufficient period for testing sorption and solubility, this test was conducted over a 14-day period. However, it should be noted that, because the specimens in this study were exposed to large amounts of distilled water, the osmotic effect would have enhanced the water sorption and solubility of specimens to a greater degree than might be found in any clinical situation (i.e. periapical tissue) [13].

The calcium silicate-based root canal sealer iRoot SP, which contains calcium phosphate, calcium hydroxide and zirconium oxide in addition to calcium silicate, has only recently become available on the market and limited data is available with regard to its sealing and solubility characteristics. Under the conditions of the present study, iRoot SP showed more water sorption than Sealapex and AH Plus. This finding may be related to the high hydrophilicity of calcium silicate, which could have allowed an ingress of water and is in agreement with previous studies [13–15] reporting materials based on MTA, the main component of which is calcium silicate, to have high levels of water uptake. Calcium hydroxide, which is formed during the setting process of both Sealapex and iRoot SP, will subsequently dissociate into calcium and hydroxyl ions [16,17], increasing both sorption and solubility while at the same time increasing pH, thereby triggering speedy repair [18–20]. In clinical situations, iRoot SP absorbs water from dentinal tubules in the canal walls to expand laterally and adopt the canal shape. As water absorption induces expansion of the material, it improves the seal between the material and dentin and makes these materials more resistant to microleakage. On the other hand, higher water uptake values may also increase the porosity of the material, which might make the material itself more susceptible to leakage. However, microleakage through dental materials has not been previously reported.

Table IV. Post-hoc comparison of sealant apical leakage.

Materials	Sealapex *Z* (*p*)	EndoREZ *Z* (*p*)	AH Plus *Z* (*p*)
iRoot SP	5.419 (*<0.001*)	5.419 (*<0.001*)	0.515 (0.620)
Sealapex		4.227 (*<0.001*)	5.417 (*<0.001*)
EndoREZ			5.391 (*<0.001*)

p-values of different groups are shown in italics.

According to ANSI/ADA Specification No.57 [11], solubility ratios of root canal sealers should not exceed 3%. This study found the solubility of the calcium hydroxide-based Sealapex in water to slightly exceed this recommended maximum and to be significantly higher than all other sealers tested at all exposure times tested. These findings are in line with those of previous reports [3] that have shown Sealapex, once set, to have a poorly formed matrix and a very high degree of solubility [10]. The high solubility of Sealapex found in the present study may be explained by the fact that the continuing ingress of water prolongs the reaction between powder and binder [10], thereby liberating additional Ca and OH ions and further increasing the porosity and thus the solubility of Sealapex over time.

In the present study, among the four sealers tested, AH plus showed the slowest gain in mass over time. This can be attributed largely to the interaction between water and the system's adsorption sites (i.e. hydroxyl group, amino group, polymer chain end), which fills the gaps between polymer chains [21]. The polymerization of AH Plus occurs through the action of its polyamine monomers (1-adamantane amine, N,N'-dibenzyl-5-oxanonandiamine-1,9, TCD-Diamine). The curing rate, cross-link density and morphology of epoxy resin is modified by the aliphatic cycle of the amine groups, which form covalent bonds with the epoxide groups when the resin's diepoxide and polyamine pastes are mixed together. Each amine group has the capacity to react with an epoxide group, resulting in a heavily cross-linked polymer with a high degree of strength and rigidity [21]. This may explain the low water sorption and solubility of AH Plus found in the present study. In a study by Schafer and Zandbiglari [3] comparing the solubility of epoxy resin (AH Plus, AH 26), silicone- (RoekoSeal), calcium hydroxide- (Apexit, Sealapex), zinc-oxide-eugenol- (Aptal-Harz) and glass ionomer-based (Ketac Endo) sealers in water and artificial saliva, AH Plus exhibited the smallest amount of weight loss of all sealers tested, independent of solvent. However, due to the lack of information regarding the chemical composition of AH Plus sealer, the authors were unable to explain the mechanism behind its low solubility [3]; however, it may be related to the sealer's resin matrix, which is more resistant to solubility.

With regard to water sorption, EndoREZ showed the greatest water sorption of all materials tested in the present study. This is a matter of concern, given that the plasticizing of a resinous matrix via water sorption and diffusion has been shown to initiate and expedite leaching of unreacted monomers [22]. The diffusion of water into the resin matrix can lead to rapid deterioration of the physico-mechanical properties of the resin, compromising the durability of resin-dentin bonds through hydrolysis and the formation of microcracks [22]. This was demonstrated in a study by Donnelly et al. [23], who observed significantly higher water sorption and solubility in EndoREZ when compared to AH Plus. While this high water sorption of EndoREZ is in concurrence to the present study, solubility rates of these two studies are different from each other. This difference can be explained by increased sample sizes and extended test periods of the present study.

Leakage continues to be a major reason for failure in root canal therapy. Although the clinical relevance of *in vitro* leakage studies has been discussed, their importance cannot be ignored, particularly in view of the scarcity of evidence-based clinical outcome studies on calcium silicate-based sealers. With regard to the measurement of microleakage, this study used an endodontic fluid transport model proposed by Wu et al. [7] that offers several advantages over more commonly used techniques for assessing leakage. The major advantage of the fluid transport model is the ability to provide a quantitative measurement of microleakage over time without destruction of experimental specimens. In addition, the sensitivity of this system can be adjusted by altering the pressure used and altering the diameter of the micropipette [7]. To avoid anatomical variations and to obtain standardization for the leakage measurements, the length of specimens was kept identical in this study. The canal diameter and anatomy were controlled to reduce study variability.

Although the manufacturers of iRoot SP claim that the calcium silicate-based sealer forms a hermetic seal inside the root canal and can be used as a filler either with or without gutta-percha points, the present study found no significant differences between the apical leakage of iRoot SP and AH Plus. This is in line with Zhang et al. [24], who also found similar leakage rates between iRoot SP and AH Plus. In this study, iRoot SP and AH Plus were found to be superior to Sealapex and EndoREZ in terms of apical leakage. Many studies have reported that AH Plus possesses better sealing properties than both Sealapex and EndoREZ [4,25]. These results correlate with other studies that have shown epoxy resin-based sealers to exhibit very little shrinkage in setting as well as dimensional stability over the long-term [3,25].

The present study found Sealapex to have the highest levels of infiltration. This finding was in contrast to that of Waltimo et al. [26], which reported good results with this sealer. Another study by Wu et al. [27] showed Sealapex to possess good initial sealing ability, but to lose this ability after long-term storage in water. The poor sealing ability of Sealapex exhibited in the present study may be related to its dissolution, correlating with previous studies [10,25]. In line with this observation, it may be suggested that high solubility is a determinant of microleakage.

The poor sealing ability of EndoREZ found in the present study is in line with the findings of several earlier studies [28,29]. Gillespie et al. [5] found polymerization shrinkage to result in gap formation between EndoREZ sealer and the root canal wall, which could explain the significantly higher degree of fluid penetration of the EndoREZ specimens when compared to the AH Plus and iRoot SP observed in the present study. Pashley et al. [30] have suggested that resin sealer tags may be torn from dentin tubules during polymerization shrinkage, creating gaps in the sealer–dentin interface that reduce sealing ability. It is possible that heat generation during searing of the sealer from the canal orifices with a heat source could expedite setting, defeating the purpose of the delayed polymerization mechanisms incorporated into the system, namely, relieving polymerization stress by slow flow [31]. Similarly, the immediate light-curing of the coronal part of the root filling, as recommended by the manufacturer to create a coronal seal, might also limit the flow of resin sealant and increase polymerization shrinkage [5]. Manipulation of the partially polymerized sealer during compaction of root-filling material may also disrupt the developing bonds between a self-etching primer and radicular dentin [32]. Finally, despite one study showing the EndoREZ system to provide a better apical seal than gutta-percha/conventional sealers by producing a 'monoblock' effect [33], the chemical link between the EndoREZ sealer and gutta-percha resin coating has been shown to be very weak [31] and the extremely high C-factor encountered in long, narrow root canals [32] suggests the creation of a monoblock within a canal would be extremely difficult.

Conclusion

The results of this study showed that all tested sealers except Sealapex met the ANSI/ADA's requirements for solubility and the calcium silicate-based sealer was not superior to other groups of sealers in terms of water sorption. No difference was found between AH Plus and iRoot SP in terms of apical sealing ability.

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