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Solubility of Endoseal and AH26 Root Canal Sealers

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Abstract

Background and Aim: Solubility of endodontic sealers should not exceed 3wt% in 24 hours. High solubility of sealers leads to gap formation and leakage through the canal wall and root filling material interface. This study aimed to compare the solubility of Endoseal and AH26 endodontic sealers.

Materials and Methods: In this in vitro, experimental study, 10 specimens with the same dimensions were fabricated from Endoseal and AH26 sealers (n=5 from each). After complete setting and initial weighing, each specimen was placed in a separate container containing 50 cc of distilled water and incubated at 37°C. The weight loss of specimens was calculated using a digital scale after 1, 3, 7, and 30 days of immersion. Data were analyzed by SPSS version 23 using t-test and repeated measures ANOVA (alpha=0.05).

Results: The overall mean weight of the two groups at different time points was not significantly different (P=0.453). Nevertheless, a significant weight change was observed in both the AH26 and Endoseal groups over time (P=0.0001). The solubility of AH26 increased while the solubility of Endoseal decreased with time (P=0.0001).

Conclusion: The solubility of both sealers was within the acceptable range according to the ANSI/ADA in the first 24 hours after setting. Both sealers experienced significant weight change over time, which was ascending in AH26 and descending in Endoseal.

Key Words: Epoxy Resin AH-26; Root Canal Therapy; Solubility

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Introduction

The primary goal of root canal therapy is to seal the root canal space to ensure a successful outcome. Endodontic sealers are imperative for three-dimensional obturation of the root canal system to create a hermetic seal and prevent the leakage of bacteria and their toxins into the periradicular tissue [1]. Root filling materials should have optimal properties such as adequate dimensional stability, insolubility, easy application, optimal biocompatibility, sealability, and radiopacity [2].

Insolubility is an important prerequisite for endodontic sealers to ensure a durable and hermetic seal after root canal obturation with gutta-percha and sealer. High solubility of sealers leads to gap formation at the root canal wall-root filling interface, and results in progressive bacterial leakage over time [3]. Thus, low solubility is a necessary prerequisite for endodontic sealers according to ISO6876 [4-6]. Accordingly, endodontic sealers should not lose more than 3wt% of their mass after immersion in distilled water for 24 hours. Moreover, release of constituents from soluble sealers can have adverse biological consequences for the periapical tissues [7, 8]. Thus, optimal biocompatibility of sealers and root filling materials is critical since they are in direct contact with the periapical tissue for long periods of time.

Calcium hydroxide-based bioceramic sealers were recently introduced to the market to improve sealing of the root canal system [9]. They have favorable properties such as complete sealing of the root canal space, and no irritation of periapical tissue. Moreover, they have bioactive and antimicrobial properties. The therapeutic effects of bioceramic sealers depend on presence of calcium hydroxide in ionized form in the periapical tissue. Thus, such sealers must possess a certain degree of solubility in the periapical tissue fluid and have the ability to release calcium hydroxide [9-11]. Evidence shows that higher amounts of released calcium ions indicate strong alkalinity [12, 13]. Long-term alkalinity confirms increased solubility of bioceramic sealers over time [14]. However, this property, if confirmed, is in contrast to the necessary prerequisite of low solubility for standardization of sealers.

Literature is controversial regarding the solubility of different endodontic sealers. AH 26, which was initially developed by Schroder in 1957 as a root canal filling material, has emerged as the most commonly employed resin sealer due to its optimal properties [15, 16]. These properties include a prolonged working time, minimal solubility, reduced shrinkage, favorable biocompatibility and antimicrobial activity, easy mixing, and superior ability to penetrate and fill the canal [15, 16]. Endoseal MTA is conveniently pre-mixed and ready to use, eliminating the need for manual mixing [9, 11, 16]. Recent studies reported several advantages for Endoseal such as fast initial setting time, minimal discoloration, optimal biocompatibility, induction of dentin remineralization, low cytotoxicity, favorable antibacterial effects, and effective penetration into dentinal tubules [9, 11, 16].

Insolubility of root canal sealers plays a critical role in minimizing microleakage and ultimately contributing to the success of root canal therapy [12, 14]. Thus, this study aimed to compare the solubility of Endoseal bioceramic sealer and the commonly used AH26 resin sealer over time.

Materials and Methods

This in vitro, experimental study was conducted on AH26 (Dentsply Sirona, PA, ISA) and Endoseal (Prevest DenPro Limited,Jammu, Kashmir) endodontic sealers. The sample size was calculated to be 5 in each of the two groups (a total of 10) according to a previous study [12], assuming alpha=5%, study power of 80%, and standard deviation of weight loss to be 0.4 to detect a significant difference equal to minimally 0.7 units in the mean weight loss between the two groups. The study protocol was approved by the ethics committee of Yazd Shahid Sadoughi University of Medical Sciences (IR.SSU.REC. 1400.132).

AH26 and Endoseal sealers were first prepared according to the manufacturers' instructions [17]. Ring molds were used to fabricate specimens with 5 mm diameter and 2 mm height from the sealers. Five specimens were fabricated from each sealer. The specimens were remained in the molds for 24 hours to completely set. After final setting, the initial weight of the specimens was measured by an electronic scale (A&D Weighing EJ-2000, Cole-Parmer, USA) with 0.0001 g accuracy. After setting of the sealers, excess material was removed, and equal dimensions of specimens were ensured for the purpose of standardization. After 24 hours, each specimen was individually immersed in 50 mL of distilled water in a plastic container [12]. All containers were coded and incubated at 37°C to simulate the oral environment for 24 hours. After 24 hours of incubation, the specimens were placed on absorbent paper and after 1 hour at room temperature, they were weighed again [12]. To ensure maximum precision, each specimen was weighed twice with a 15-minute interval. This process was repeated after 3, 7 and 30 days of immersion. the weight of AH26 and Endoseal specimens was measured again after one month of storage in dry environment following day 30 of immersion to ensure complete dehydration of specimens. Weight loss compared with the initial weight of specimens indicated their solubility. The following equation was used to calculate the solubility percentage of the sealers [12]:

$$\text{Solubility (\%)} = \frac{W_0 - W_f}{W_0} \times 100$$

Where W0 indicates the initial weight of specimen and W1 indicates its weight at each measurement time point.

Data were analyzed using SPSS version 22 by t-test and repeated measures ANOVA at P<0.05 level of significance.

Results

Table 1 presents the mean weight of AH26 and Endoseal specimens at different time points and its percentage of change over time. According to t-test, the mean initial weight of AH26 and Endoseal specimens was not significantly different (P=0.521).

The mean weight of AH26 and Endoseal specimens was not significantly different at 1 day (P=0.915), 3 days (P=0.516), 7 days (P=0.289), or 30 days (P=0.116) either. As shown, the overall mean weight of the two groups at all time points was not significantly different between the two groups (P=0.453).

The intra-group comparisons revealed a statistically significant increase in the mean weight of both sealers on day 1 (P=0.001 for AH26 and P=0.015 for Endoseal). At 3 days, the mean weight of AH26 specimens significantly increased compared with their initial weight (it is worth mentioning that incomplete desiccation process can affect the weighing results) (P=0.0001). However, the increase in the mean weight of Endoseal specimens compared with their initial weight was not significant (P=0.072). At 7 days, the mean weight of AH26 specimens significantly increased compared with their initial weight (P=0.0001).

Table 1. Mean weight of AH26 and Endoseal specimens at different time points and its percentage of change over time (mg)

Time point of measurement	Sealer	Initial weight mean± SD *	Weight at the mentioned time point mean± SD	Weight change mean± SD	Percentage of weight change	P-value **
	AH26	22±238	22±245	1±6	2.94	0.001
Day 1	Endoseal	5±231	6±246	3±14	6.49	0.015
	P-value	0.521	0.915	-	-	-
	AH26	22±238	22±246	1±7	3.36	0.0001
Day 3	Endoseal	5±231	7±239	3±7	3.46	0.072
	P-value.	0521	0.516	-	-	-
	AH26	22±238	22±246	0.1±7	3.36	0.0001
Day 7	Endoseal	5±231	7±236	4±4	2.16	0.123
	P-value	0.521	0.289	-	-	-
	AH26	22±238	20±250	1±11	5.04	0.004
Day 30	Endoseal	5±231	4±230	6±0.8-	0.4	0.688
	P-value	0.521	0.116	-	-	-

* SD: Standard deviation

** T-test

However, the increase in the mean weight of Endoseal specimens compared with their initial weight was not significant (P=0.289). At 30 days, the mean weight of AH26 specimens significantly increased compared with their initial weight (P=0.004). Nonetheless, a reduction in the mean weight of Endoseal specimens was observed in comparison with their initial weight; however, this reduction did not reach statistical significance (P=0.688). Repeated measures ANOVA demonstrated a significant weight change in both groups over time (P=0.0001). The solubility of AH26 had an ascending trend while that of Endoseal had a descending trend over time (Figure 1). The trend of weight change was also significantly different between the two groups over time (P=0.0001).

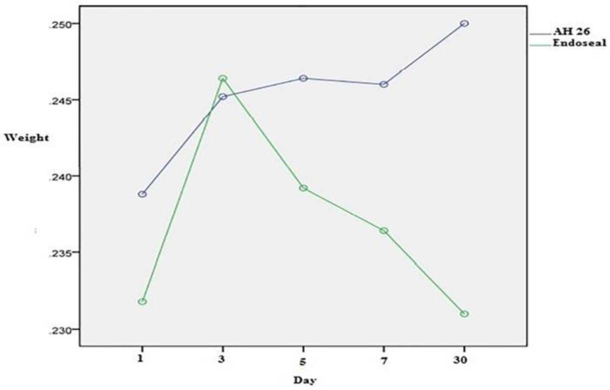

Figure 1. Trend of weight change of AH26 and Endoseal specimens over time

Discussion

Absence of apical seal is the most important cause of endodontic treatment failure [18]. According to Grossman, an ideal sealer should not be soluble in tissue fluids in order to be able to preserve the apical seal over time [19]. This in vitro study compared the solubility of AH26 and Endoseal endodontic sealers.

In the current study, distilled water was used as the solvent because of the fact that water is the primary constituent of both intracellular and extracellular fluids [3, 20]. However, it is worth noting that weak acids have been used in some studies as solvent for this particular purpose [21].

Two methods are often adopted by in vitro studies for assessment of solubility of dental materials [3]. The first method is to immerse the material in a solvent and then quantify the amount of released ions into the solvent by atomic absorption spectrometry, to finally determine the solubility of the respective material. In the second method, which was adopted in the present study, the specimens fabricated of the respective material are immersed in a solvent, and their weight loss over time is quantified by using a digital scale. This method was also used by some studies because it is low-cost and does not require advanced equipment [14, 22-24]. In the present study, the specimens were weighed after 1, 3, 7, and 30 days. According to the ANSI/ADA and ISO-6876, a maximum of 3wt% of mass change after 24 hours of water immersion is acceptable for a sealer [4-6].

The present results showed that the weight gain of AH26 sealer after 1 day was 2.94%, which was within the acceptable range by the ANSI/ADA specification number 30 and 57, and ISO-6876. However, the percentage of weight gain increased with time, such that it reached 5.04% at 30 days, indicating high water sorption. Ashraf et al. [22] compared the physical properties and chemical characterization of two experimental epoxy resin root canal sealers (ES-A and ES-B) with AH-26. The solubility of ES-A, ES-B, and AH-26 sealers was found to be 0.0053%, 0.0051%, and 0.0048%, respectively, after 24 hours. Statistical analysis revealed no significant difference in solubility among the tested resin-based sealers. It is noteworthy that variations in the surface-to-volume ratio of specimens, as well as differences in experimental configurations such as the molds used and setting time, might have contributed to the variations in the reported results.

Zordan-Bronzel et al. [23] compared the solubility of Bio-C sealer and TotalFill BC sealer with AH Plus. After 30 days, AH Plus showed the lowest solubility (0.2%), followed by TotalFill BC sealer (10.6%) and Bio-C sealer (17.9%); the difference in solubility was significant among the three sealers. Urban et al. [24] compared the solubility of BioRoot RCS and MTA Fillapex with AH Plus. The highest solubility was found in MTA Fillapex followed by BioRoot RCS; AH Plus showed the lowest solubility and the difference in solubility was statistically significant among the sealers.

Lim et al. [25] compared the solubility of Endoseal sealer with MTA and AH Plus. Endoseal showed the highest solubility among the tested materials although there was no significant difference among the three groups. Saavedra et al. [26] compared AH Plus, GuttaFlow Bioseal, Endoseal MTA, Bio-C sealer and BioRoot RCS. GuttaFlow Bioseal showed the lowest solubility followed by AH Plus; the remaining three sealers did not meet the ISO6876:2012 solubility requirements, and the observed difference was statistically significant.

Notably, conventional solubility tests do not simulate the clinical setting and may overestimate the solubility of hydrophilic sealers, such as calcium silicate-based sealers [9, 23]. Also, the final drying process in the conventional tests may cause water evaporation and consequent loss in total mass [27] which may explain why some calcium silicate-based sealers failed to meet the ISO6876:2012 standards in some of the aforementioned studies. Difference in the reported values in the aforementioned studies and the present study may be due to different sizes of specimens, because the surface/mass ratio can affect the solubility of materials. In the present study, AH26 sealer had a weight gain of 0.42% at 7 days. The weight gain of AH26 observed in the present study is due to its high water sorption, which can compensate for its weight loss due to solubility. Thus, the weight of AH26 specimens was measured again after one month of storage in a dry environment following day 30 of immersion to ensure complete dehydration of specimens; the results showed 231.2 mg weight (compared with 238 mg initial weight after setting). This finding indicated a reduction in pure mass of specimens. Hence, notwithstanding the apparent weight gain observed in humid conditions, AH26 specimens experienced 7 mg weight loss, which was offset by water sorption and thus remained undetectable. The weight of Endoseal specimens was measured to be 226 mg after one month of dry storage following 30 days of immersion in water. Thus, the pure mass loss of AH26 was greater than that of Endoseal.

Some review studies [28, 29] mentioned the low solubility of Endoseal in tissue fluids as an advantage. The present results revealed that the weight change of Endoseal bioceramic sealer was +6.49% at day 1. Over time, it experienced a gradual weight loss, resulting in 0.4% reduction at 30 days, in comparison with its initial weight. The authors conducted a comprehensive literature search and found no study regarding the solubility of Endoseal over time. However, some studies assessed the solubility of other bioceramic sealers. For instance, Poggio et al. [14] showed that BioRoot™RCS and TotalFill BC Sealer had significantly higher solubility among the tested materials, and experienced a weight change > 3%. Viapiana et al. [30] reported high solubility of MTA-Fillapex. Lee et al. [31] discussed that incomplete setting of EndoSequence BC and MTA-Fillapex sealers even 1 month after humid incubation was the reason for their higher solubility. Controversy in the reported results can be attributed to different methodologies such as techniques of drying of specimens after solubility testing.

The present results revealed that Endoseal had high water sorption at 1 day, which resulted in its significant weight gain. Over time, it lost weight, and solubility of this sealer had a descending trend such that its weight at 30 days was lower than its baseline weight. This study evaluated the specimens for only 30 days. Evaluation for longer periods of time could have revealed further weight loss and dissolution, which calls for further studies with longer follow-ups.

Bioceramic cements and sealers often release calcium hydroxide. The released calcium may induce cementum formation due to its bioactivity, and stop the process of dissolution. In the present study, the mean weight of Endoseal specimens was lower than that of AH26 at all time points but not significantly. However, the trend of weight change in both groups was significant over time, and was ascending in AH26, and descending in Endoseal.

Two factors affect the solubility of sealers in contact with body fluids. The first factor is hydrolysis, which refers to loss of sealer content by water solubility, and eventually results in weight loss and reduction of sealer content. The second factor is water sorption by sealer, which acts in contrast to the first factor, and results in weight gain [3, 32,33]. Water sorption, especially by AH26, was a confounding factor in assessment of its solubility in the present study. Moreover, the observed weight loss may not be entirely due to solubility. The filler particles present in sealer composition may be dislodged and released without dissolution. Such confounding factors complicate the precise assessment of solubility of materials [3]. To overcome this problem, the type of ions released from the sealer into the solvent can be identified by atomic absorption spectrometry to reveal the true solubility percentage of material. The size of specimens is another important factor to consider since surface/mass ratio affects solubility [3]. Thus, the size of specimens should be standardized to obtain accurate results.

This study had some limitations, such as the large surface area of specimens (much larger than the surface area of apically extruded sealers in clinical scenarios), and short duration of study (evaluation of specimens for only 30 days). Future studies on a higher number of specimens over longer periods of time are recommended. Also, atomic absorption spectrometry should be used in future studies to obtain more accurate results regarding the solubility of sealers.

Conclusion

The solubility of both sealers was within the accepted range by the ANSI/ADA in the first 24 hours after setting. Both sealers experienced significant weight change over time, which was ascending in AH26 and descending in Endoseal.

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