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A four-week solubility assessment of AH-26 and four new root canal sealers

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ABSTRACT

Background: The strong link between sealer solubility and periapical reinfection indicates that water solubility of new sealers should be studied. This study aimed to assess the water solubility of five root canal sealers (AH-26, Topseal, 2-Seal, Acroseal, and Roeko Seal Automix [RSA]).

Materials and Methods: In this *in vitro* experimental study, 30 specimens were fabricated from each of the abovementioned sealants. Then they were weighed and randomly divided into three subgroups of 10 each (A, B, and C). They were set at 37°C and 100% RH, in accordance with ANSI/ADA 57 and ISO 6876-2001 requirements. Afterward, the specimens in subgroups A were incubated at 37°C and 100% RH for 24 hours, while the specimens in the subgroups B and C were incubated in the same conditions for 7 days and 28 days, respectively. After incubation, the specimens were dried with blotting paper and were incubated for 24 hours at 37°C and 0% RH. Then they were weighed. The percentage of weight loss was regarded as water solubility.

Results: The mean solubility of the sealers AH-26, Acroseal, Topseal, 2-Seal, and RSA were 0.28%, 0.36%, 0.07%, 0.037%, and 0.141% after 24 hours, respectively. After 28 days, their solubility were 1.75%, 0.746%, 0.082%, 0.04%, and 0.517%, respectively. Only the solubility of the sealers 2-Seal and Topseal were not statistically different ($P>0.3$ [Tukey's]). Again only the solubility of 2-Seal and Topseal did not significantly increase between the 7th day and the 28th day of incubation ($P>0.6$ [paired-samples *t*]).

Conclusion: All tested materials met the standards (maximum weight loss of 3% within 24 hours). However, the results of 2-Seal followed by Topseal were the most favorable ones.

Key Words: Dental materials, endodontics, root canal sealants, solubility, standards

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INTRODUCTION

Obturation materials are used in root canal therapy (RCT) to entomb the residual microorganisms or their toxins, fill the inaccessible areas, and seal the canal in order to prevent coronal leakage which is a major cause of RCT failure.[1-8] The obturation mainly depends on the sealers to prevent ingress of microorganisms from the oral environment and their passage to the periapical areas. Degradation of the sealer may cause gaps at dentin/sealer or gutta-percha/sealer junctions, which can facilitate bacterial proliferation and colonization.[2,9-11] Therefore, low water solubility of sealers has a major impact on success, longevity, and prognosis of RCT.[8,9,11,12]

The quality of the seal obtained with gutta-percha and conventional zinc oxide eugenol (ZOE) sealers is not perfect.[3,9] Hence, several new resin cement sealants have been developed to be used instead of ZOE and to improve the root canal seal beyond that currently possible with conventional materials.[1-3,10,13] These include silicon-based sealers

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which are well tolerated by tissues and have low water sorption,[1,2,10,14] calcium hydroxide-based sealers which are highly antibacterial and may partly denature toxic proteins,[1,2,5,10] as well as epoxy resin-based sealers with the possibility of adhesion to dentin and lower rates of water solubility.[1-3,6,8,10,13,15] Nevertheless, calcium hydroxide-based sealers are highly soluble,[5] and resin-based and silicon-based materials are also soluble which may endanger a proper seal; although the solubility of resin-based materials is usually lesser than that of ZOE (which is reported as between 1% and 7%)[9,12,16] and does not exceed a maximum weight loss of 3% within 24 hours of distilled water storage (in accordance with the standards for RCT sealers [ANSI/ADA No. 57 and ISO 6876-2001]).[1-3,8,10,13]

The strong link between sealer solubility and periapical reinfection indicates that water solubility of new sealers should be studied.[13] However, surprisingly few studies have been carried out in this matter.[9] To our knowledge the solubility of some new sealers has not been assessed before, such as two epoxy resin sealers (TopSeal [Dentsply Maillefer, Ballaigues, Switzerland] and 2-Seal [VDW, Endodontic Synergy, Munchen, Germany]). In addition, there are only few studies with regard to the solubility of some other sealants such as a calcium hydroxide-based sealer (Acroseal [Septodent, France])[8] and a silicon-based one [polydimethyl syloxane] (RoekoSeal Automix [Roeko, Langenau, Germany]).[9,17] Thus the aim of this study was to comparatively evaluate the water solubility of the above-mentioned sealers and that of a well-known epoxy resin-based sealer (AH-26 [Dentsply]).

MATERIALS AND METHODS

This experimental study was performed on 150 specimens fabricated with the mentioned 5 brands of root canal sealers: AH-26, Topseal, 2-Seal, Acroseal, and RSA. For preparing each specimen, a cylindrical copper mold 3 mm high and 5 mm in diameter was used.[12] After cleaning the molds with acetone and then with distilled water, each mold was weighed three times with a degree of accuracy of 0.0001 g (Mettler, College, Germany) and the average weight was recorded.[9,12] Afterward, the molds were randomly divided into five groups of 30 specimens each.

The molds in each group were filled with one of the experimental materials which were prepared according to the manufacturers' instructions. Afterward, the specimens were set by incubation in 100% relative humidity at 37°C. Incubation times were predetermined according to the manufacturers: AH-26: 15 hours, Topseal: 8 hours, Roeko Seal Automix (RSA): 50 min, 2-Seal: 8 hours, Acroseal: 24 hours.

Solubility assessment

After incubation, each specimen was weighed for three times with the digital scale and the mean weight was calculated. Afterward, each group was randomly divided into three subgroups of ten each. The subgroups were A: 24-hour incubation; B: 7 days incubation; and C: 28 days incubation.

The specimens in each subgroup were stored in a sealed container of double-distilled water. All the containers were placed in the incubator at 37°C with 0% humidity. After 24 hours, the subgroups A were removed from the incubator, after 1 week the subgroups B were removed, and after 4 weeks the subgroups C were removed for solubility inspection. After removing each subgroup, the specimens were taken out of the container and were dried with blotting papers. Then they were incubated in dry air at 37°C for another 24 hours to become absolutely dried.

Afterward, each specimen was weighed for three times and the average weight was recorded. The percentage of solubility was calculated according to the following formula: Solubility (%) = $((W_0 - W_f) / W_0) \times 100$, in which W_0 and W_f were initial and final weights (g), respectively.[4,9,11,12]

Statistical analysis

Descriptive statistics were calculated. A one-way analysis of variance (ANOVA), a Tukey's *post hoc* test, and a paired-samples *t* test were used to analyze the data. The level of significance was set at 0.05.

RESULTS

The one-way ANOVA showed that there were significant differences between the experimental materials after 24 hours (P=0.000), after 7 days (P=0.000), and after 28 days of incubation (P=0.000, [Table 1], [Figure 1]). Tukey's test showed that only the mean solubility values of Topseal and 2-Seal materials had no significant differences after either 24 hours (P=0.510), or 7 days (P=0.307), or 28 days (P=0.646). All the other materials showed significant differences with each other at all intervals (all P values <0.02).

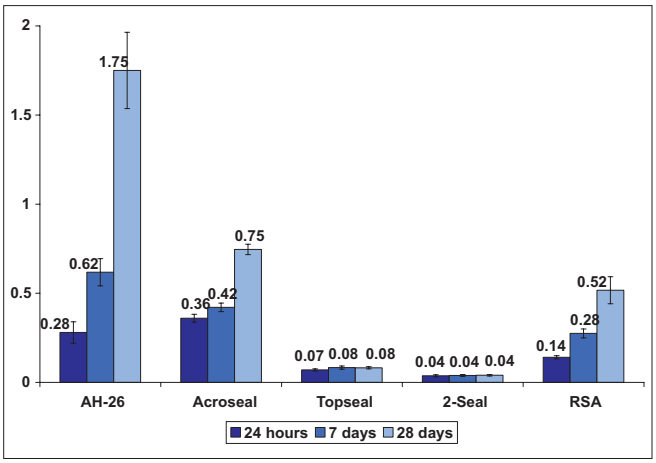

Figure 1: Mean (SD) of solubility percentages at different testing times

Table 1: Mean solubility (±standard deviation) of the experimental groups after different periods of incubation (in percentage)

Time	Sealers	Mean (%)	SD (%)	CV (%)	95% CI	
					Low	Up
24 hours	AH-26	0.28	0.060	21.4	0.243	0.317
	Acroseal	0.36	0.022	6.1	0.346	0.374
	Topseal	0.07	0.006	8.6	0.066	0.074
	2-Seal	0.037	0.006	16.2	0.033	0.041
	RSA	0.141	0.009	6.4	0.135	0.147
7 days	AH-26	0.618	0.076	12.3	0.571	0.665
	Acroseal	0.421	0.024	5.7	0.406	0.436
	Topseal	0.083	0.009	10.8	0.077	0.089
	2-Seal	0.039	0.005	12.8	0.036	0.042
	RSA	0.275	0.025	9.1	0.260	0.290
28 days	AH-26	1.75	0.214	12.2	1.617	1.883
	Acroseal	0.746	0.029	3.9	0.728	0.764
	Topseal	0.082	0.006	7.3	0.078	0.086
	2-Seal	0.04	0.004	10.0	0.038	0.042
	RSA	0.517	0.076	14.7	0.470	0.564

RSA: Roeko Seal Automix, SD: Standard deviation, CV: Coefficient of variation, CI: Confidence interval

The comparison between the solubility values of the subgroups A with the subgroups C (using the paired-samples *t* test) showed that only the sealer 2-Seal did not have a significant increase in solubility (P=0.255) and that the other ones showed significant increases (P=0.000). The comparison between the subgroups A and B showed that again only the sealer 2-Seal did not show any significant increases (P=0.445), while the other sealers had significant increases in the subgroups B compared with A (P=0.000). Comparing the mean solubility of the subgroups B and C showed that the sealers 2-Seal (P=0.702) and Topseal (P=0.634) did not increase after the 7th day, and the others increased significantly from the 7th day (B) to the 28th day (C) (P=0.000).

DISCUSSION

In the present study, the procedures were performed as outlined in the International Standard ISO 6876 (2001).[9] Each specimen was immersed in double-distilled water only once, in order to improve the accuracy of the measurements by avoiding the undesirable weight loss of the sealer due to repeated immersions and dryings.[9] Furthermore, to our knowledge all other studies set the sealers in uniform conditions,[7,9,12] while it might not comply with the manufacturers' different instructions for different brands. Thus in this study, the setting time was determined exactly according to the manufacturers to reproduce *in vivo* conditions. The low coefficients of variation calculated may indicate low sample dispersion and thus high reproducibility, consistency, and reliability of the methods used.

All the tested groups in this study showed solubility rates within the acceptable range (3% weight loss) required by ANSI/ADA No. 57 and ISO 6876 (2001) as well as British Standard BS 6934 (1998),[12] which was in line with the results of other studies.[6,9,17,18] However three of the experimental materials showed an increasing trend which seemed to have a possibility to exceed that level in delayed course. The solubility of the two newly tested epoxy resin-based sealers favorably ceased to increase, and their results were statistically similar.

Under the conditions of this study, two of the epoxy resin-based sealers showed the best results compared with the calcium hydroxide- and silicon-based sealers, which this finding was comparable to the results of testing another epoxy resin material (AH-Plus)[4,9,17] and supported the findings of other studies.[4,6,7,9,12,15,18,19] After 24 hours of incubation, the calcium hydroxide-based sealer showed the highest solubility rate. In agreement with the findings of Shafer and Zandbiglari,[9] its result was poorer than that of the epoxy resins and the silicon. However, after 28 days, the solubility of other calcium hydroxide-based brands used in their study[9] was two to four times greater than that of the calcium hydroxide sealer examined in this study, which might be due to the level of hydrophobic materials in the sealers that can block the ingress of water.[9] The purpose of adding calcium hydroxide to the sealer is to maintain health or promote the healing procedure of periapical tissues as well as its antimicrobial effects. Nonetheless, dissociation of calcium and hydroxyl ions out of the

sealer is necessary for providing its tissue healing effects. Such a mechanism is in contrast to the philosophy of sealants, and may compromise coronal seal in long term.[1,5,9,10,20] However, solubility of one of the epoxy resin-based sealers (AH-26) surpassed that of the calcium hydroxide-based material at the 28th day. Moreover, similar to the study of Shafer and Zandbiglari,[9] after 28 days RSA showed better results compared with AH-26. The AH-26 used in this study revealed a solubility approximately half of what was observed in their study.[9] The initial solubility of AH-26 was low, which was consistent with other studies,[6,9,18] and might be explained by relative insolubility of its additives.[6,9] However, again similar to the previous studies,[9,18] some solubility was noted on the 28th day. This phenomenon and also the higher rate of solubility observed in this material might be attributable to degradation of unreacted hexamethylenetetramine polymer and its breakdown to ammonia and formaldehyde.[9,18] It should be taken into consideration that the differences in surface-to-volume values of the specimens as well as other experimental configurations such as molds used and setting times might contribute to the differences in the results.

The present experiment was limited by some factors. We measured the elution of water-soluble materials, but not the actual solubility which is defined as the thermodynamic equilibrium of a pure chemical compound with the solution,[9,21] although this method was consistent with the standards. Moreover filler disintegration during immersion and evaporation of volatile sealer components during drying procedures might also cause some weight loss.[6,9,21] Furthermore, water sorption might affect the weight loss, although it might be noticeable mostly in glass ionomer and ZOE sealers.[6,9,18] It has been suggested that in order to reproduce tissue fluids and oral environment, media such as culture medium, artificial saliva or dilute acid should be used for solubility tests rather than distilled water.[6,9,15,21] However, artificial saliva does not necessarily resemble oral conditions,[6] and distilled water usage is required by the standards. Moreover, according to the manufacturer of AH-26, a setting time of 15 hours was sufficient to set the AH-26 and we as well as Shafer and Zandbiglari[9] could not find any evidence of a partially set material. Even they[9] stated that it needed at least 1 week for setting.

CONCLUSION

All the sealers met the standards for RCT sealers. However, two of the epoxy resin-based ones (especially the 2-Seal brand) were consistently of the lowest solubility over time. The brands ranked in order of favorable long-term solubility were as follows: 2-Seal, Topseal, RSA, Acroseal, and AH-26.

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