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RESEARCH ARTICLE OPEN ACCESS

Solubility, Flowability, and Setting Time of a New Endodontic Sealer based on Polycaprolactone

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Abstract:

Aim: One of the essential materials required for root canal treatment is a sealer. This study aimed to assess the solubility, flowability, and setting time of a new endodontic sealer compared to a commercial sealer, AH Plus.

Material and Methods: In this *in vitro* study, the new endodontic sealer was prepared using various proportions of the materials and compared with the control group (AH Plus sealer). Standard methods were used to measure solubility, flowability, and setting time. Statistical analysis was performed using t-tests and GraphPad Prism 9 software.

Results: The new sealer demonstrated lower solubility, higher flowability, and longer setting time compared to AH Plus.

Conclusion: Given the favorable results of the new sealer in this study, it is recommended for use in future clinical studies.

Keywords: Endodontic sealer, Solubility, Setting time, Flowability, AH Plus sealer, Polycaprolactone.

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1. INTRODUCTION

In dentistry, root canal treatment is performed to fill the root canal space and eliminate intra-canal complications. For successful treatment, the canal must be adequately filled, and all infectious microorganisms must be eradicated. One of the essential materials required for root canal treatment is a sealer. Sealers fill the spaces between gutta-percha and canal walls effectively, creating a suitable seal to prevent colonization of oral microorganisms in periapical tissues and within the canal space [1]. A good sealer should possess characteristics, such as flowability, radiopacity, compatibility with oral tissues, antimicrobial properties, adequate solubility, appropriate working time, non-toxicity, dimensional stability, and adequate adhesion to canal components [2].

In the year 2000, according to the new amendments of the American National Standards Institute (ANSI) and the American Dental Association (ADA), new tests and criteria were introduced to evaluate the physicochemical properties of dental sealers for standardization and enhancement of research quality. These tests included film thickness, setting time, flowability, radiopacity, solubility, and dimensional changes [3].

AH26 sealer is an epoxy resin widely used as a sealer. It exhibits good flowability, effectively seals dentinal walls, and has sufficient working time. Like many sealers, AH26 is highly toxic when freshly prepared, but its toxicity decreases over time. Previous research has shown that the toxicity of AH26 is mainly due to the release of formaldehyde. However, recent studies using modern analytical methods indicate that the mechanism of cellular toxicity of the sealer is due to the activation of COX-2 in RNA gene expression, leading to reduced expression of inducible Nitric Oxide Synthase (iNOS) protein and synergistic effects on root resorption. Canal sealers comprising Lipopolysaccharide (LPS) induce COX-2 expression in macrophage cells. Toxicity may rapidly decrease during setting and become less toxic after 24 h. The new formula of AH26, AH Plus, is a dual-paste system that is claimed not to release free formaldehyde during setting. It has greater radiopacity, shorter setting time (approximately 8 hours), lower solubility, and better flowability compared to AH26. A study has shown that AH Plus has less short-term and long-term toxicity and is less genotoxic compared to AH26 [4].

There are various types of sealers with different bases, including Zinc Oxide Eugenol (ZOE), calcium hydroxide, glass ionomer, epoxy resin, silicone-based, and bioceramic sealers [5]. Among them, AH Plus is used as the gold standard among other endodontic sealers [6]. However, it has been reported that this sealer does not have adequate adhesion to canal walls in the presence of moisture. In a study comparing the solubility of several sealers (EasySeal, MTA Fillapex, Pulp Canal Sealer, Sealapex, AH Plus, and N2) conducted by Poggio *et al.*, AH Plus showed very low solubility (0.01-0.045%), being acceptable according to international standards [7]. Additionally, a study by Almeida *et al*. demonstrated AH Plus to have a flowability equivalent to 32.8 ± 1.7 mm according to ISO 6876 standards, an initial setting time of 8 ± 1.2 h, and a final setting time of 15.2 ± 1.6 h, according to ASTM-C266-08 standards, passing all relevant standards [8].

Resin-based sealers are the most common and well-known sealers in contemporary endodontics, suitable for both lateral and vertical techniques. In the past, ZOE-based sealers have been more prevalent. Drug-based sealers (such as Cortone) and calcium hydroxide-based sealers, glass ionomer, *etc*., have been introduced in the past but have never been as reliable and popular as resin-based sealers in terms of study results and commercial reliability. Polycaprolactone is a biocompatible synthetic polymer widely used in medical, pharmaceutical, and dental applications. The resin of this material is considered an excellent base for producing novel resins in endodontics [9].

The reason for using polycaprolactone in the sealer is that it is resistant to dissolution in water, biodegradable, has antimicrobial properties, and possesses relatively good mechanical properties for use in root canal filling during root canal treatment [10].

Solubility, flowability, and setting time are special properties of sealers used in dentistry for root canal repair. Despite the importance of this issue, few studies have been conducted on the solubility of endodontic sealers. Limited studies on various types of sealers have shown different results [10]. Adequate flowability and setting time are also crucial for proper clinical performance and should be proportionate to the working time [8]. Therefore, the aim of this study was to investigate the solubility, flowability, and setting time of a new endodontic sealer and compare them with those properties of AH Plus sealer.

2. MATERIALS AND METHODS

This study was conducted on 18 sealer disc samples, divided into 2 groups: 9 samples of the new sealer and 9 samples of the commercial AH Plus sealer used as the control. The sample size was determined based on the study by Lee *et al*. [11], and considering the standard deviation for the variables, a type I error of 0.05, and a power of 80%, the number of samples in each group was determined.

2.1. Preparation of the New Endodontic Sealer

To prepare the sealer, bioactive glass (45% SiO_2, 24.5% Na2O, 24.5% CaO, and 6% P_2O_5) powders (21.5%), zinc oxide (21.5%), and barium sulfate (22%) were mixed using the spatulation method. The mixing was continued until a uniform mixture was achieved. Additionally, the mixture was sonicated in an ultrasonic bath for 1 h to ensure homogeneity. Polycaprolactone resin (P767 and P787) was heated to 70 ºC, with 25% and 10% by weight, respectively, and the remaining powders were dissolved in it to completely combine with the polycaprolactone to form a homogeneous paste.

Table **1** shows a comparison of the ingredients of the experimental *vs*. the control sealers.

Table 1. The main ingredients of the experimental and the control sealers.

Group Name	Ingredients			
Experimental group: the new sealer	Bioactive glass (45% SiO_2, 24.5% Na_2O, 24.5% CaO, and 6% P_2O_5)	Zinc oxide	Barium sulfate	Poly-caprolactone resin (a mixture of P767 and P787)
Control group: AH Plus sealer	Diepoxide	Calcium tungstate	Zirconium oxide	Aerosil

2.2. The Solubility Test

The cylindrical copper molds with a height of 3 mm and a diameter of 5 mm were used (Fig. **1**). The molds were cleaned with acetone and distilled water and then weighed. Their weights were recorded. The sealer samples in both groups were then prepared using these molds. After preparation, the samples were incubated at 100% humidity and 37 ºC for 8 h (the incubation time was according to the protocol of the commercial sealer manufacturer, cited in the sources as 8 hours for AH Plus). After the incubation period, the samples were weighed three times, and the average weight was noted.

Fig. (1). Steps for evaluating the solubility of a sealer.

The samples from each group were then divided into three subgroups, each with a different incubation period at 37 ºC and 0% humidity.

The subgroups were incubated as follows:

- Subgroup 1: 1 day
- Subgroup 2: 7 days
- Subgroup 3: 28 days

After the incubation period, the samples were removed, blotted with paper to remove moisture, and placed in an oven to dry completely. The samples were then carefully weighed three times, and the average weight was recorded.

Solubility was determined according to ISO 4049 standards (11). The solubility of the samples was calculated using the following formula, where W_0 is the original mass of the sample and W_r is the removed mass of the sample (11).

Solubility (%) = $(W_r / W_0) \times 100$

2.3. Flowability Test

This study was conducted according to ISO 6875 standards (11). For testing the flowability, 0.5 ml of sealer was mixed for 3 min, and a 20 g plate (±2 g error) with a 100 g weight was placed on the center of the sealer. After 10 min, the plate was removed and the diameter of the spread sealer was measured using calipers or a digital caliper [12].

2.4. Setting Time Test

This test was also conducted according to ISO 6875 standards under a temperature of 37±1 ºC and a humidity of 95±5%. In this test, three cylinders with a diameter of 10 mm and a thickness of 2 mm were prepared from the mixed sealer [according to the standards of the American Society for Testing and Materials (ASTM)]. Then, using the initial Gilmore needle (113.4 g), marks were made on the sample every 60 sec. The setting time was recorded when the needle was no longer able to make a mark, indicating the initial setting time. The final setting time was determined similarly using the final Gilmore needle (453.6 g) [8].

2.5. Statistical Analysis

Results have been reported using descriptive statistical indices. A t-test was used to compare the setting time between the two groups. GraphPad 9 software was used for data analysis. A significance level of less than 0.05 was considered as a significance level.

Table 2. The results of the comparison of solubility between the two groups.

Solubility	AH Plus	New Sealer
1	137	122
2	139	135
3	136	127
4	135	120
5	136	131
6	133	129
7	134	128
8	137	129
9	132	132
Mean	135.44	128.11
SD	2.06	4.43

3. RESULTS

The results showed the new sealer compared to the commercial sealer AH Plus to have lower solubility (p=0.0005), higher flowability (p=0.0001), longer initial setting time (p=0.0001), and longer secondary setting time (p=0.0001).

Table **2** presents the results of the comparison of solubility between the two groups. The mean solubility for the new sealer was 128.11±4.43, and for the commercial sealer, it was 135.44±2.06.

Table **3** displays the results of the comparison of flowability between the two study groups. The average flowability for the new sealer was 57.04±2.026 mm, while for the AH Plus sealer, it was 47.03±1.27 mm.

Table 3. The results concerning the comparison of flowability between the two study groups.

Flowability	AH Plus	New Sealer
1	50.05	54.5
2	46.17	58.3
3	46.01	56.24
4	47.2	56.2
5	48	60.4
6	46.25	61
7	48.6	53.8
8	45.3	54.3
9	45.7	58.7
Mean	47.03	57.04
SD	1.27	2.026

Table **4** displays the results related to the comparison of initial setting time between the two study groups. The mean initial setting time for the new sealer was 284.66±16.8, while for the commercial sealer, it was 206.33±11.5 mm.

Table 4. The results related to the comparison of initial setting time between the two study groups.

Setting Time 1	AH Plus	New Sealer
1	230	245
2	221	274
3	189	259
4	195	300
5	220	299
6	200	286
7	205	296
8	199	301
9	198	302
Mean	206.33	284.66
SD	11.5	16.8

Table **5** presents the results related to the comparison of secondary setting time between the two study groups. The mean secondary setting time for the new sealer was 696.55±14.9 min, while for the commercial sealer, it was 640.44±30.17 min.

Table 5. The results related to the comparison of secondary setting time between the two study groups.

Setting Time 2	AH Plus	New Sealer
1	660	682
2	623	701
3	689	689
4	585	745
5	645	698
6	666	701
7	615	705
8	603	654
9	678	694
Mean	640.44	696.55
SD	30.17	14.9

4. DISCUSSION

Solubility, flowability, and setting time are essential properties of sealers used in endodontic root canal treatment. The results have demonstrated the new sealer, compared to the commercial sealer AH Plus, to have lower solubility (p=0.0005), higher flow (p=0.0001), longer initial setting time (p=0.0001), and longer secondary setting time (p=0.0001).

In the present study, the average solubility for the new sealer was 128.11±4.43, and for the commercial sealer, it was 135.44±2.06. According to standard no. 57 of the American Dental Association, endodontic sealers' solubility should not exceed 3% of their weight [13] after being immersed in distilled water for 24 hours, which both sealers examined in this study met. Additionally, the new sealer exhibited lower solubility (p=0.0005) compared to the commercial sealer AH Plus. Certainly, the structure of a material and its constituent substances play a significant role in its solubility. The use of water-resistant materials or materials with low solubility in the structure of a sealer, as well as the use of absorbent materials, can reduce its solubility [14]. Given the uniformity of all conditions for the new sealer and the commercial sealer in the solubility study, it can be considered that differences in their structures in terms of constituent materials could have affected the lower solubility of the produced sealer. The sealer produced in this study was composed of bioactive glass (21.5%), zinc oxide (21.5%), barium sulfate (22%), and poly-caprolactone resin (a mixture of P767 and P787), all of which are insoluble in water except for bioactive glass.

In fact, low solubility is one of the most important characteristics of materials used as sealers in endodontic treatments [7].

Shokriamzad *et al*. investigated the solubility of zinc oxide eugenol (ZOE), Dorifill, AH26, and Tubliseal sealers in distilled water. The results indicated that Dorifill sealer demonstrated the highest solubility among the studied sealers with a 2.8% weight loss over 7 days. Tubliseal and ZOE sealers showed weight losses of 1.4% and 2.3%, respectively. AH26 sealer, on the other hand, showed a 1.9% weight gain over the same period. The average weight change among the ZOE, Dorifill, and Tubliseal groups did not have a statistically significant difference (p>0.05). However, a significant statistical difference was observed between the average weight change of AH26 sealer and the other groups (p<0.001). The authors stated that since the overall solubility percentage of all examined sealers fell within the standard range determined for the solubility level of sealers, they could be considered clinically approved for use based on this physical property [15].

In this study, the comparison of flow rates between the two study groups showed the mean flow rate for the new sealer to be 57.04±2.26, and for the commercial sealer, it was 47.03±1.27 millimeters. The results indicated that both sealers met the ADA requirements for the flow rate of an endodontic sealer based on the measured drop sizes

(d> 20 millimeters) [16]. Adjusting the size and shape of particles in the structure of a sealer can help improve flowability [17]. The presence of zinc oxide particles in the material structure can contribute to this [17]. Given the uniform conditions for both the new and commercial sealers in the flow rate study, the reason for the better flowability of the new sealer compared to the commercial sealer may be attributed to the size and morphology of the powder used in their structure [17].

Alik *et al*. examined the flow rate of three ZOE-based endodontic sealers in various consistencies. The experimental group included prepared samples of the sealers as follows: a) endomethasone at ratios of liquid to powder, *i.e*., 1:5, 1:6, 1:7 (standard), 1:8, and 1:9, according to the manufacturer's brochure; b) Roth 801 as a mixture of 1:7 (standard) and 1:8; and c) Tubliseal EWT as a standard preparation (base-catalyst 1:1). A volume of 0.5 mm of sealer was dispensed onto a glass slide, and the sealer diameter was measured by applying a 2-kilogram load. Based on the measured drop sizes of the sealers, all of them met the ADA requirements for appropriate flowability (d> 20 mm) [18].

In this study, the mean initial setting time for the new sealer was 284.66±16.8 min, and for the commercial sealer, it was 206.33±11.5 min. Additionally, the mean secondary setting time for the new sealer was 14.9±696.55 min, and for the commercial sealer, it was 640.44±30.17 min. Both sealers met the standard setting time of 9-12 hours for an endodontic sealer [16].

Koo *et al*. compared the setting time of four sealers (CeraSeal, EndoSeal TCS, One-Fil, and Well-Root) with the AH Plus sealer. The sealer samples were kept in an incubator at 95% humidity and 37 ºC. A Gillmore needle with a total weight of 100 g and a diameter of 2.0 mm was precisely placed vertically against the sealer, and the setting time was recorded when the needle no longer created an indentation on the sealer surface. AH Plus exhibited the longest setting time. EndoSeal TCS, One-Fil, and CeraSeal showed the shortest setting times when using gypsum molds among the five types of sealers. The authors concluded that the selected sealers for setting require moisture. Lack of moisture leads to a significant delay in setting time. Since root canals contain moisture, it is necessary to test the setting time of the various sealers using gypsum molds to determine the biological status of the root canals [16].

In our previous studies, we have prepared and assessed the cell biocompatibility and sealing ability of polycaprolactone-based endodontics sealer in comparison to AH Plus sealer [19, 20]. The results for cell biocompatibility have shown AH Plus to exhibit moderate cytotoxic effects against dental pulp stem cells (the number of living cells was 49.45 ± 10.56%). However, for the new sealer, the number of living cells was ≥ 80% (81% ± 4.11) [19]. Thus, the sample was non-cytotoxic against the tested cells. It also showed a sealing effect similar to AH Plus as a commercial sealer [20]. Then, the new sealer presented in this study is recommended for use in future clinical studies.

CONCLUSION

All three properties examined (solubility, flowability, and setting time) for the new sealer in this study have been found to be satisfactory and within the standard range. This new material is recommended for use in dental clinical studies. It is necessary to evaluate the mechanical properties and adhesion of the new sealer in the near future.

AUTHORS' CONTRIBUTION

It is hereby acknowledged that all authors have accepted responsibility for the manuscript's content and consented to its submission. They have meticulously reviewed all results and unanimously approved the final version of the manuscript.

LIST OF ABBREVIATIONS

ANSI	=	American National Standards Institute
ADA	=	American Dental Association
iNOS	=	Inducible nitric oxide synthase
LPS	=	lipopolysaccharide
ZOE	=	Zinc oxide eugenol

ETHICS APPROVAL AND CONSENT TO PARTICIPATE

Not applicable.

HUMAN AND ANIMAL RIGHTS

Not applicable.

CONSENT FOR PUBLICATION

Not applicable.

AVAILABILITY OF DATA AND MATERIAL

All data generated or analyzed during this study are included in this published article.

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CONFLICT OF INTEREST

Simin Sharifi is the editorial advisory board member of the journal TODENTJ.

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