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ORIGINAL ARTICLE

Evaluating the Solubility of Endodontic Sealers in Response to Static and Dynamic Stress: An *In Vitro* Study

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ABSTRACT

Objective: This study investigated the effect of static and dynamic conditions on the solubility of three endodontic sealers: AH Plus (an epoxy-resin-based sealer), Bio-C Sealer (a bioceramic sealer), and EndoSequence BC (a bioceramic sealer).

Methods: Plastic moulds were used to create 150 specimens, which were divided into three groups, with each group being filled with one of the three sealers. After the setting time, the specimens were removed from the moulds, dried, and weighed. Thirty specimens from each group were subdivided into three subgroups and stored in distilled water for 30, 60, or 90 days, while the remaining 20 specimens in each group were subdivided into four subgroups and subjected to 0, 20, 50, or 100 thermocycling cycles. After storage time and thermocycling, the specimens were reweighed, and the weight loss and solubility percentage were calculated. The data were analysed using one-way ANOVA, Post Hoc Tukey, and Pearson correlation tests ($p<0.05$).

Results: The results showed that AH Plus had the lowest solubility, followed by EndoSequence BC and then Bio-C Sealer ($p<0.0001$). Both static and dynamic conditions had a negative effect on the solubility of all tested sealers, with the effect being more pronounced in bioceramic sealers. The increase in storage days and the number of thermocycling cycles were significantly correlated with the increasing solubility levels of all tested sealers ($p<0.0001$).

Conclusion: The increased solubility of endodontic sealers may have a negative impact on long-term treatment outcomes. Both static and dynamic conditions can affect the solubility of endodontic sealers.

Keywords: AH Plus, bioceramic sealer, root canal sealer, solubility, thermocycling

HIGHLIGHTS

- The solubility of AH Plus was found to be the lowest, followed by EndoSequence and then Bio-C Sealer.
- Both static and dynamic conditions can negatively affect the solubility of endodontic sealers.
- Bioceramic sealers, such as Bio-C Sealers, are more susceptible to the negative effects of static and dynamic conditions on solubility than other sealers.

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INTRODUCTION

In root canal therapy (RCT), the main goal is to disinfect and obturate the three-dimensional root canal space (RCS) using endodontic sealers in combination with gutta-percha (1, 2). The ideal sealer should have biocompatibility, slow setting time, appropriate working time, good sealing ability, adhesion to root canal walls, dimensional stability, low solubility, and good radiodensity (3). High sealer solubility can lead to chemical release that causes inflammatory reactions and gap formation in the RCS, which increases the risk of bacterial leakage and treatment failure (4–6). The American Dental Association (ADA) recommends less than 3% solubility for root canal sealers to maintain their sealing ability and prevent reinfection.

Root canal sealers are classified based on composition, including resin, calcium hydroxide, mineral trioxide aggregate (MTA), and glass ionomer. AH Plus(AHP), an epoxy resin-based sealer, is considered the gold standard sealer due to its physicochemical properties but lacks bioactivity (7, 8). Therefore, other endodontic sealers, such as bioceramic sealers, have recently gained wide attention because of their formation of apatite layer deposition. It was claimed that this layer is a tag-like structure capable of bonding with dentine and providing better sealing ability than other sealers (9). Although currently used endodontic sealers meet the ADA solubility requirements, it is still being determined if they maintain solubility in different static and dynamic situations (10–13).

Thermocycling is a reliable method to artificially age dental biomaterials (14–16). It involves subjecting the material to temperature changes ranging from 5°C to 55°C, simulating thermal stresses (17). It was shown that 20–50 cycles of thermocycling are equivalent to one day of environmental changes in the oral cavity (18). Previously, thermocycling was used to determine the effect of artificial ageing on the apical sealing ability of endodontic sealers (19).

Few studies have been carried out on the solubility of root canal sealers in response to dynamic stress. The present study intended to evaluate the effect of artificial ageing on the solubility of AHP, Bio-C Sealer (BCS), and EndoSequence BC Sealer (ESS). The null hypothesis was that the static and dynamic conditions did not increase the solubility of AHP (epoxy resin-based sealer) and the tested BCS and ESS.

MATERIALS AND METHODS

Sample Size Calculation

G*Power software (University of Dusseldorf, Dusseldorf, Germany) was used to determine the sample size. Considering an alpha-type error of 0.05 and a power of 95% (effect size=0.324), 50 specimens were estimated for each group (AH Plus (AHP), Bio-C (BCS), and EndoSequence BC (ESS)). Thirty specimens from each group were subdivided into three subgroups according to the storage time in distilled water: 30, 60, or 90 days (n=10/subgroup). The remaining 20 specimens from each group were subdivided into four subgroups according to the thermocycling cycles: 0, 20, 50, or 100 (n=5/subgroup). This study did not involve humans or biological materials taken from them. Therefore, ethics committee approval form was not obtained.

Specimen Preparation

One hundred and fifty circular polycarbonate moulds (Falcon Plastics, Div. of BioQuest, Oxnard, CA) measuring 1.5 mm high ×7.75 mm in diameter were purchased. Thirty specimens from each of the three groups were dedicated to evaluating the effect of storage on solubility. The remaining 20 specimens in each group were dedicated to evaluating the effect of thermocycling on solubility. Moulds were divided into three groups based on the type of sealer: i) AH Plus (Dentsply-Malleifer, Ballaigues, Switzerland), ii) Bio-C Sealer (Bio-C Sealer; Angelus, PR, Brazil), and iii) EndoSequence BC (Brasseler USA, Savannah, GA, USA) (Table 1). Sealers were mixed according to the manufacturer's instructions and placed inside the tubes using a mixing spatula. The specimens were placed between 2 glass plates covered with cellophane film. All sealers except for AHP, which required moisture for setting, were assessed by placing two pieces of wet cloth between the mould and the glass plates. All specimens were placed in an incubator at 37°C and 100% humidity for 72 hours. The test specimens were removed from the moulds, kept in a desiccator, and weighed on a precision balance (0.001 g professional mini scale, T-series, Insten, China).

Storage Process

Once the sealers were set, thirty specimens from each of the three groups were subdivided into three subgroups of 10 (n=10) based on the storage days. The moulds were stored in plastic flasks containing 7.5 mL distilled water and kept in an oven (Carbolite Gero, United Kingdom) at 37°C for either 30, 60, or 90 days.

Thermocycling Procedure

Twenty specimens in each group were subdivided into four subgroups of 5 (n=5). Then, specimens were subjected to thermocycling for either 0 cycles (control), 20 cycles, 50 cycles, or 100 cycles according to the method described previously by Saghiri et al. (14–16). The thermocycling process was done using a programmed robot (TeachMover; Microbot, Questech, Inc.) that transferred the specimens between 2 temperature-controlled water baths. Four beakers, two large (300 mL) and two small (20 mL) were used. One large and one small beaker served as cold baths, while the other set of large and small beakers served as warm baths. Both large and small beakers were filled with normal saline, and the small ones were placed inside the larger beakers. Thermocycling included transferring specimens between cold baths (5°C) (small beaker) and warm baths (55°C) (small beaker), with a dwell time of 30 seconds in each bath and a transfer time of 15 seconds between each bath.

Solubility

The solubility assessment was performed using the method used by Carvalho-Junior et al. (4) and Saghiri et al. (20–22). After storage time and each thermocycling process, the specimens were placed in a desiccator and reweighed until the mass stabilised to obtain their final weights. The percentage of solubility was calculated as follows: (IM-FM)/IM×100 (where IM is the initial mass and FM is the final mass of the specimen after storage or thermocycling process).

Statistical Analysis

Data were analysed using Kolmogorov-Smirnov, one-way ANOVA, Pearson correlation, and Post Hoc Tukey tests at the level of significance $p<0.05$. All statistical analyses were done using SPSS statistical software (version 25, SPSS, Chicago, IL).

TABLE 1. The name, manufacturer, and composition of endodontic sealers

Material	Manufacturer	Composition
AH Plus	Dentsply-Malleifer, Ballaigues, Switzerland	Bisphenol-A epoxy resin, bisphenol-F epoxy resin, calcium tungstate, zirconium oxide, silica, iron oxide pigments dibenzyldiamine, aminoadamantane, silicone oil
Bio-C Sealer	Angelus, Londrina, PR, Brazil	Calcium silicates, calcium oxide, calcium aluminate, zirconium oxide, iron oxide, silicon dioxide, dispersing agent
Endosequence BC Sealer	Brasseler USA, Savannah, GA, USA	Zirconium oxide, calcium silicates, calcium phosphate monobasic, calcium hydroxide, filler, and thickening agents

AH: An epoxy-resin-based sealer; BC: Bio-C; PR: Parana; USA: United States of America ; GA: Georgia

TABLE 2. The means and standard deviations of solubility (%) for the tested materials at three different storage times (30, 60, and 90 days)

Storage time	AH Plus	Bio-C Sealer	Endosequence BC Sealer
30 days	0.41±0.11%	15.91±2.21%	7.12±0.82%
60 days	0.86±0.13%	34.02±4.78%	15.88±1.87%
90 days	1.18±0.12%	48.87±3.58%	22.03±1.44%

AH: An epoxy-resin-based sealer; BC: Bio-C

TABLE 3. The means and standard deviations of solubility (%) for the tested endodontic sealers at three different cycles (0, 20, 50, and 100)

Thermocycling cycles	AH Plus	Bio-C Sealer	Endosequence BC Sealer
0 cycles	0.30±0.05%	7.22±1.37%	3.56±0.53%
20 cycles	1.04±0.21%	15.38±1.93%	6.14±1.33%
50 cycles	5.40±1.00%	33.42±3.56%	17.94±1.22%
100 cycles	7.74±0.68%	53.00±3.39%	34.34±2.22%

AH: An epoxy-resin-based sealer; BC: Bio-C

RESULTS

The one sample Kolmogorov-Smirnov test showed a normal distribution of data in values. The one-way ANOVA test showed significant differences between and among experimental groups of both storage and thermocycling subgroups ($p<0.0001$). The Post Hoc Tukey test was used to determine differences between the solubility percentages between thermocycling and storage subgroups. Significant differences were detected between subgroups within each group ($p<0.0001$) (Fig. 1a).

The means and standard deviations of solubility (%) for the tested materials at three different storage times (30, 60, and 90 days) are shown in Table 2. The lowest solubility values were seen in the AHP group, and the highest were in the BCS group. In each group, the solubility percentage was increased with increasing storage days. The means and standard deviations of resultant values in experimental groups are shown in Table 3. The lowest solubility values were seen in the AHP group, and the highest were in the BCS group. In each group, the solubility percentage increased with increasing thermocycling cycles.

Among the specimens subjected to thermocycling, significant differences were seen between solubility values after 20, 50, and 100 cycles ($p<0.0001$) (Fig. 1b). In comparison, there were no significant differences between values of 0 and 20 cycles within AHP and ESS groups ($p>0.05$).

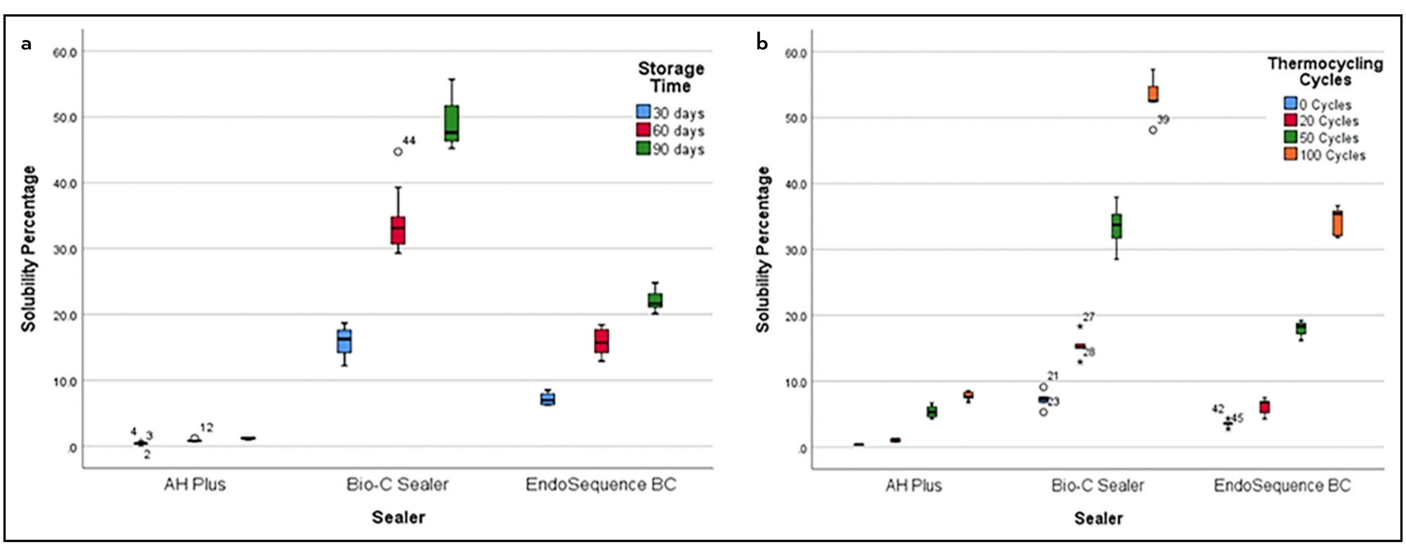

Figure 1. (a) Box plots of means and standard deviations of solubility (%) for the tested endodontic sealers at three different storage times (30, 60, and 90 days). (b) Box plots and standard deviations of solubility (%) of tested endodontic sealers for the tested endodontic sealers after thermocycling (0, 20, 50, and 100 cycles)

AH: An epoxy-resin-based sealer; BC: Bio-C

The Pearson correlation showed a significant correlation between the solubility and storage days in all groups (p<0.0001). Similarly, a significant correlation was detected between solubility and thermocycling cycles in all groups (p<0.0001). Increasing storage days or thermocycling cycles increased the solubility of all tested sealers (p<0.0001).

DISCUSSION

The null hypothesis was rejected as the static and dynamic conditions negatively affected the solubility of the tested sealers. The results of the present study showed that the solubility of endodontic sealers increased with increasing storage days. The highest solubility percentage was seen after 90 days of storage in distilled water. The comparison between the experimental groups indicated that the specimens of the AHP group had the lowest percentage of solubility compared with the BCS and ESS groups. These results were in accordance with previous studies, where AHP showed the least amount of solubility and weight loss compared with other tested endodontic sealers (23, 24). The lower solubility of AHP might be explained by the numerous cross-links present in this epoxy resin-based sealer's structure, making it less soluble in distilled water (25). Zordan-Bronzel et al. (23) evaluated the solubility of endodontic sealers after 30 days of immersion in distilled water and reported similar results, as the percentage of solubility of BCS was higher than AHP after 30 days.

Similarly, Zhou et al. (26) reported that ESS had a higher solubility when compared with AHP. In addition, the results of the present study indicated that BCS had significantly higher solubility between the tested bioceramic sealers when compared with the ESS group. After 90 days, BCS lost 48.87% of its weight compared with ESS, which only decreased by 22.03%.

The evaluation of the solubility of sealers after thermocycling showed similar outcomes, whereas tested endodontic sealers after 20, 50, and 100 cycles showed a higher percentage of solubility. BCS showed the highest solubility, followed by ESS and AHP sealers. When comparing the solubility of bioceramic sealers, BCS showed much higher solubility percentages when compared with ESS. After 100 cycles, BCS had a solubility of 53% compared with ESS, which had a solubility of 34.34%. As mentioned previously, thermocycling is a reliable method to simulate the stresses of dynamic situations within the oral cavity (12, 14, 16). The adverse effect of thermocycling on the properties of endodontic sealers has been discussed previously (and are) in accordance with the results of the present study (19). Lin et al. (19) showed that the apical sealing ability of tested endodontic sealers decreased as the number of thermocycles increased.

Another point that might be considered in similar studies can be the medium in which the tested endodontic sealers have been stored. For instance, the authors used distilled water as the storage medium in the present study. However, in previous studies, some authors used a variety of mediums such as phosphate-buffered solution, simulated body fluid, and Hank's Balanced Salt Solution (Sigma-Aldrich, Inc. Missouri, US) (27–29). For future studies, investigations should be performed to evaluate the effect of different storage mediums on the physical properties of root canal sealers.

One of the strengths of this study was the innovative approach to testing the effect of thermal stresses on root canal sealers. The present study is the first to investigate the effect of thermal shocks and stresses on endodontic sealers. This study had some limitations, one of which was using only three types of endodontic sealers, limiting the generalisability of the findings to other sealers. Additionally, the study focused only on the solubility of sealers, which may not provide a comprehensive understanding of the sealers' effectiveness.

Future directions for research involve investigating a more comprehensive range of endodontic sealers and evaluating other physicochemical properties such as biocompatibility, mechanical properties, antibacterial properties, and physicochemical properties. Long-term clinical studies could also be conducted to evaluate the impact of sealer solubility on the success and durability of endodontic treatments.

CONCLUSION

Based on the outcomes of the present study, it was concluded that dynamic conditions have greater negative effects on the solubility and mass loss of endodontic sealers compared to the storage of sealers in distilled water. Bioceramic sealers, including BCS and ESS, have much higher solubility percentage and mass loss than the AHP under static and dynamic conditions. These findings underscore the importance of considering the potential impact of static and dynamic stresses on endodontic sealers when selecting and using them in clinical practice, mainly when using bioceramic sealers.

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