

Original Article

Comparative Evaluation of Solubility and Calcium Ion Release of Bioceramic and Resin-Based Root Canal Sealers

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This study aimed to evaluate and compare the solubility and calcium ion release of four root canal sealers: Endosequence (bioceramic), BioRoot RCS (bioceramic), MTA Fillapex (tricalcium silicate-based), and AH Plus (resin-based). Polyethylene tubes were filled with the respective sealers and tested for solubility by measuring weight loss after immersion in distilled water, and for calcium ion release using an atomic absorption spectrophotometer. Intergroup comparisons were conducted using one-way ANOVA with post hoc Bonferroni tests, while intragroup comparisons between time periods were analyzed using repeated measures ANOVA with post hoc Bonferroni tests. The results showed significant differences in solubility and calcium ion release among the tested sealers. MTA Fillapex exhibited the highest solubility, ranging from 12.79 ± 0.17 μm on Day 1 to 19.85 ± 0.14 μm on Day 28, while AH Plus had the lowest, with values from -1.05 ± 0.72 μm to -2.32 ± 0.27 μm . In terms of calcium ion release, Endosequence Bioceramic sealer demonstrated the highest release, ranging from 10.01 ± 0.22 mg/L on Day 1 to 10.59 ± 0.51 mg/L on Day 28, whereas AH Plus had the lowest release, from 1.98 ± 0.32 mg/L to 0.54 ± 0.13 mg/L . The bioceramic sealer- Endosequence, demonstrated favorable physicochemical properties in terms of low solubility and sustained calcium ion release compared to the other sealers tested. These findings suggest that the bioceramic sealers may be advantageous for improving the long-term success of endodontic treatment.

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Introduction

The primary objective of endodontic treatment is to eliminate infection, prevent further disease progression, and facilitate the restoration of the affected tooth to its proper function. At the core of successful endodontic therapy lies the achievement of a proper three-dimensional seal at the root apex [1]. Inadequately sealed root canal systems can harbor residual microbial flora, which is a major contributing factor to endodontic treatment failures, accounting for up to 58% of cases [2]. Microleakage, often caused by poor adaptation between the sealer and the gutta-percha or the dentin, as well as the presence of voids within the sealer, is a primary reason for endodontic treatment failure [3]. The use of an effective root canal sealer that can fill irregularities and penetrate into the dentinal tubules is essential to ensure a durable and hermetic seal.

In 1936, Grossman described the ideal properties of a root canal sealer, which include providing an excellent hermetic seal, adequate adhesion to the root canal walls, dimensional stability, radiopacity, biocompatibility, and lack of staining [4]. Over the years, various types of root canal sealers have been developed, each with its own set of physicochemical and biological characteristics. Traditionally, zinc oxide-eugenol-based sealers were commonly used in endodontic practice. However, these sealers were found to exhibit poor adhesion to dentin and cause discoloration of the tooth structure [5]. The introduction of resin-based sealers, such as AH Plus (DentsplySirona, York, PA), addressed some of these drawbacks. AH Plus, an epoxy resin-based sealer, does not contain eugenol and has been widely used in clinical practice due to its good adhesion to dentin walls [6].

In recent decades, the development of bioceramic-based sealers has gained significant attention in the field of endodontics. Bioceramics are inorganic, non-metallic, and biocompatible materials with similar mechanical properties to dental hard tissues [7]. These sealers interact with hydroxyapatite crystals and form a mineral infiltration zone, exhibiting enhanced chemical stability,

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alkaline pH, and lack of shrinkage. Endosequence (Brasseler USA, Savannah, GA) is a bioceramic-based sealer that has been widely used in clinical practice. It is a premixed, injectable, and radiopaque sealer that is capable of self-setting and expansion, which can contribute to its sealing ability [8]. BioRoot RCS (Septodont, Saint-Maur-des-Fossés, France) is another bioceramic sealer that releases calcium hydroxide upon setting, potentially providing an alkaline environment within the root canal system [8].

MTA Fillapex (Angelus, Londrina, Brazil) is an inert tricalcium silicate-based sealer that does not form calcium hydroxide when it sets. In contrast to BioRoot RCS, MTA Fillapex exhibits a relatively low calcium ion release, which may impact its ability to maintain an alkaline pH within the root canal environment [9]. The physicochemical properties of root canal sealers, such as solubility and calcium ion release, are considered crucial in defining a successful obturation [10]. An ideal endodontic sealer should have low solubility to prevent dispersion in the canal system and the formation of voids [11]. Additionally, the sustained release of calcium ions from the sealer helps maintain an alkaline pH within the root canal system, which inhibits the growth of microbial flora. Maintaining an alkaline pH within the root canal system is important, as it inhibits the growth of microbial flora, which thrives in an acidic environment [12]. The presence of calcium hydroxide in the root canal environment has been shown to exert antimicrobial effects, promote the formation of a mineralized barrier, and stimulate the regeneration of periradicular tissues [13].

Several studies have investigated the solubility and calcium ion release of various root canal sealers, including bioceramic and resin-based formulations [11,14-18]. However, a comprehensive comparison of these properties among Endosequence, BioRoot RCS, MTA Fillapex, and AH Plus sealers is lacking in the current literature. The current study aimed to evaluate and compare the solubility and calcium ion release of Endosequence, BioRoot RCS, MTA Fillapex, and AH Plus root canal sealers. The findings of this investigation might provide valuable insights into the selection of an appropriate root canal sealer with favorable physicochemical properties, ultimately contributing to the long-term success of endodontic treatment.

The specific objectives of this study were:

1. To evaluate and compare the solubility of Endosequence, BioRoot RCS, MTA Fillapex, and AH Plus root canal sealers.
2. To assess and compare the calcium ion release from Endosequence, BioRoot RCS, MTA Fillapex, and AH Plus root canal sealers.
3. To correlate the solubility and calcium ion release characteristics of the tested sealers and discuss their implications for endodontic practice.

Materials and Methods

The study used polyethylene tubes as the sample containers. The tubes were 10 mm in diameter and 3 mm in height. The sealers evaluated in the study were: Endosequence (Bioceramic sealer), BioRoot RCS, MTA Fillapex and Resin sealer (AH Plus).

The polyethylene tubes were cut into smaller sections of equal dimensions (10 mm diameter, 3 mm height). The size of each sample tube was pre-measured using a digital vernier caliper. The tubes were pre-weighed using a digital weighing balance (SEPTTECH precision balance) to select tubes with similar weights. The tubes were divided into four experimental groups, with 20 tubes assigned to each group.

Sealer Placement: (a) Endosequence (Bioceramic sealer)

Endosequence is a premixed, injectable bioceramic sealer with a smooth, creamy paste-like consistency. This sealer is provided in a ready-to-use formulation and does not require any mixing. It can be directly dispensed from the syringe. The sealer is transferred by slowly extruding it from the syringe into the polyethylene tubes. The Endosequence sealer is gently packed into the tubes using a plugger or a similar instrument to ensure complete filling without voids.

Sealer Placement: (b) BioRoot RCS

BioRoot RCS is a calcium silicate-based sealer with a smooth, homogeneous paste-like consistency. The powder and liquid components of BioRoot RCS are mixed according to the manufacturer's instructions, usually in a 3:1 powder-to-liquid ratio, to obtain a creamy, smooth consistency. The mixed BioRoot RCS sealer is transferred to the polyethylene tubes using a suitable delivery instrument, such as a lentulo spiral or a syringe. The BioRoot RCS sealer is carefully packed into the tubes, ensuring complete and uniform filling.

Sealer Placement: (c) MTA Fillapex

MTA Fillapex is a salicylate resin-based sealer with a thick, putty-like consistency. The two-paste components of MTA Fillapex are mixed according to the manufacturer's instructions until a homogeneous mixture is obtained. The mixed MTA Fillapex sealer is transferred to the polyethylene tubes using a suitable delivery instrument, such as a plastic filling instrument or a syringe. The MTA Fillapex sealer is gently packed into the tubes, ensuring complete and uniform filling.

Sealer Placement: (d) Resin sealer (AH Plus)

AH Plus is an epoxy resin-based sealer with a smooth, creamy paste-like consistency. The base and catalyst components of AH Plus are mixed according to the manufacturer's instructions, usually in a 1:1 ratio, until a homogeneous mixture is obtained. The mixed AH Plus sealer is transferred to the polyethylene tubes using a suitable delivery instrument, such as a syringe or a mixing pad and a filling instrument. The AH Plus sealer is carefully packed into the tubes, ensuring complete and uniform filling without any voids.

The tubes were left in an incubator at 37°C and 100% humidity until the sealers were completely set. Each disc (sample) was tied with an impermeable nylon thread to prevent damage during weighing. The initial weight of each disc (W₀) was measured using the SEPTTECH precision balance digital machine. Each sample was immersed in 20 ml of deionized water for 1, 7, 14, and 28 days. The samples were kept in an incubator at 37°C and 100% humidity during the immersion period.

Evaluation of Solubility

After each immersion period (1, 7, 14, and 28 days), the discs were removed from the solution, dried with blotting paper, and left undisturbed for 24 hours to ensure complete drying. The discs were then weighed again using the SEPTTECH precision balance (W_{f1}, W_{f7}, W_{f14}, W_{f28}). The solubility (%) was calculated using the formula,

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

Assessment of Calcium Ion Release

After the polyethylene tube samples were immersed in deionized water for 1, 7, 14, and 28 days, the solutions containing the leached ions were collected for analysis. The atomic absorption

apetrophotometer (model AA-6300, Shimadzu) was used to quantify the amount of calcium ions released from the sealer samples into the deionized water over the different time intervals (1, 7, 14, and 28 days). Prior to the analysis, the Atomic Absorption Spectrophotometer instrument was calibrated using standard calcium solutions of known concentrations. The water samples containing the leached calcium ions from the sealer specimens were aspirated into the Atomic Absorption Spectrophotometer, and the instrument measured the absorbance of the samples at the specific wavelength for calcium (422.7 nm). The absorbance values obtained from the atomic absorption spectrophotometer were then used to calculate the concentration of calcium ions in the water samples. The calcium ion concentration was typically reported in milligrams per liter (mg/L) or parts per million (ppm).

A pilot study was conducted by using 5 samples in each group. Sample size was computed for the original study based on the results of pilot study. The study parameters included a significance level (alpha) of 0.05 and a desired power of 0.85. The expected effect size (delta) was set at 0.4106, with between-group variance (Var_m) of 1253.5 and within-group variance (Var_e) of 7435.8. Based on these parameters, the estimated total sample size required was 80 samples, with 20 participants per group, ensuring sufficient power to detect the specified effect size in the ANOVA.

The statistical analysis was performed using SPSS (Statistical package for social sciences, version 27, IBM Corp., Armonk, USA). Descriptive statistics pertaining for the assessed study variable “solubility” and “calcium ion release” in each sealer group was represented using mean and standard deviation. The normality of the data distribution pertaining to the assessed study variable was verified using Shapiro Wilk test on residuals. The equality of variances between the compared groups was verified using Bartlett’s test. Intergroup comparison for solubility and calcium ion release was performed using one-way ANOVA followed by post hoc Bonferroni test. Intragroup comparison for solubility and ion release between time periods was performed using repeated measures of ANOVA followed by post hoc Bonferroni test. For all comparisons, p<0.05 was considered to be statistically significant.

Results

Descriptive statistics pertaining for the assessed study variable

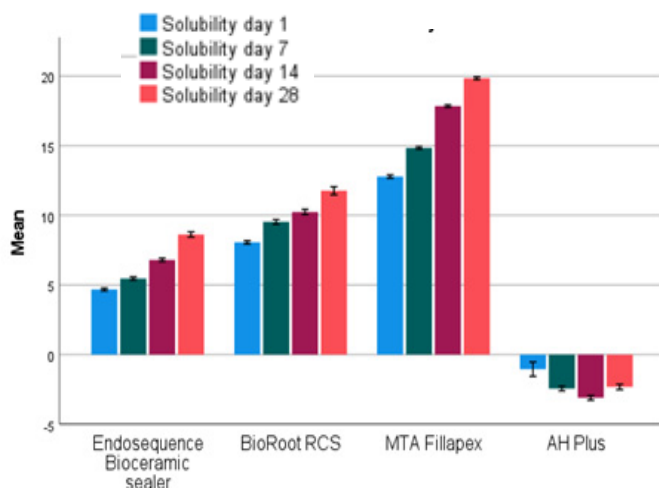


Figure 1: Mean values for solubility in different root canal sealers

“solubility” and “calcium ion release” in each sealer group was represented using mean and standard deviation (table 1 and 2). There was a statistically significant difference between the compared sealers for both solubility and calcium ion release at all time periods (day 1, day 7, day 14 and day 28) (table 1 and 2). Higher solubility values were observed for MTA Fillapex followed by BioRoot RCS, Endosequence bioceramic sealer and AH plus sealer (table 1). Greater percentage of calcium ion release was observed for Endosequence bioceramic sealer followed by MTA Fillapex, BioRoot RCS and AH plus sealer (table 2). Intragroup comparison revealed that there was statistically significant difference between the compared time periods for solubility in all sealer groups except AH Plus (table 1). There was statistically significant difference between all compared time periods for solubility in AH plus except between day 7 and day 28 (table 1). Intragroup comparison revealed that there was statistically significant difference between the compared time periods for calcium ion release in all sealer groups except AH Plus (table 2). There was statistically significant difference between all compared time periods for calcium ion release in AH plus except between day 1 and day 7 (table 2).

MTA Fillapex showed the highest solubility, ranging from $12.79 \pm 0.17 \mu\text{m}$ on day 1 to $19.85 \pm 0.14 \mu\text{m}$ on day 28, followed by BioRoot RCS, Endosequence Bioceramic sealer, and AH Plus, which had the lowest solubility ranging from $-1.05 \pm 0.72 \mu\text{m}$ on day 1 to $-2.32 \pm 0.27 \mu\text{m}$ on day 28 (table 1). In terms of calcium ion release, Endosequence Bioceramic sealer exhibited the greatest release, from $10.01 \pm 0.22 \text{ mg/L}$ on Day 1 to $10.59 \pm 0.51 \text{ mg/L}$ on Day 28, followed by MTA Fillapex, BioRoot RCS, and AH Plus, which showed the lowest release from $1.98 \pm 0.32 \text{ mg/L}$ on Day 1 to $0.54 \pm 0.13 \text{ mg/L}$ on Day 28 (table 2). Mean values for solubility and calcium ion release in different sealer groups were illustrated in figures 1 and 2.

Discussion

The results of this study provide valuable insights into the physicochemical properties of various root canal sealers, which are crucial factors in determining the long-term success of endodontic treatment. The selection of an appropriate root canal sealer is essential, as it plays a pivotal role in establishing a durable and well-sealed root canal system, which is fundamental for the prevention of endodontic treatment failure. The solubility of root canal sealers

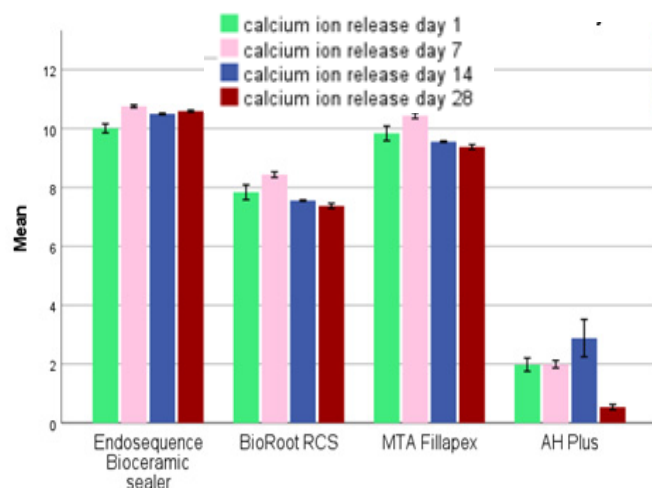


Figure 2: Mean values for calcium ion release in different root canal sealers

Table 1: Solubility values observed in root canal sealers after storage in deionized water

Sealer	Day 1	Day 7	Day 14	Day 28
EndosequenceBioceramic sealer	4.67 ± 0.14 ^{Aa}	5.46 ± 0.17 ^{Ba}	6.80 ± 0.17 ^{Ca}	8.63 ± 0.26 ^{Da}
BioRoot RCS	8.06 ± 0.17 ^{Ab}	9.53 ± 0.23 ^{Bb}	10.24 ± 0.26 ^{Cb}	11.77 ± 0.40 ^{Db}
MTA Fillapex	12.79 ± 0.17 ^{Ac}	14.84 ± 0.13 ^{Bc}	17.85 ± 0.13 ^{Cc}	19.85 ± 0.14 ^{Dc}
AH Plus	-1.05 ± 0.72 ^{Ad}	-2.44 ± 0.23 ^{Bd}	-3.10 ± 0.24 ^{Cd}	-2.32 ± 0.27 ^{Bd}

Different lower case letters in the same columns indicates statistical significance

Different upper case letters in the same rows indicates statistical significance

Same upper case letters in the same rows indicates statistical insignificance

Table 2: Calcium ion release values observed in root canal sealers after storage in deionized water

Sealer	Day 1	Day 7	Day 14	Day 28
EndosequenceBioceramic sealer	10.01 ± 0.22 ^{Aa}	10.76 ± 0.66 ^{Ba}	10.50 ± 0.37 ^{Ca}	10.59 ± 0.51 ^{Da}
BioRoot RCS	7.84 ± 0.35 ^{Ab}	8.44 ± 0.13 ^{Bb}	7.56 ± 0.31 ^{Cb}	7.37 ± 0.12 ^{Db}
MTA Fillapex	9.84 ± 0.35 ^{Ac}	10.44 ± 0.13 ^{Bc}	9.56 ± 0.31 ^{Cc}	9.37 ± 0.12 ^{Dc}
AH Plus	1.98 ± 0.32 ^{Ad}	2.00 ± 0.18 ^{Ad}	2.88 ± 0.89 ^{Cd}	0.54 ± 0.13 ^{Dd}

Different lower case letters in the same columns indicates statistical significance

Different upper case letters in the same rows indicates statistical significance

Same upper case letters in the same rows indicates statistical insignificance

is an important property, as low solubility is desirable to prevent the formation of voids and maintain the integrity of the root canal seal over time [11]. The resin sealer, AH plus exhibited the least solubility among the compared root canal sealers. The cross-linked polymeric structure of the resin matrix in AH plus provides high resistance to dissolution and disintegration when exposed to fluids in the root canal system [19]. The hydrophobic nature of the resin matrix helps repel water and reduces the penetration of aqueous fluids into the sealer, thereby limiting its solubility [19].

In the present study, the bioceramic sealer, Endosequence exhibited significantly lower solubility compared to the BioRoot RCS and the MTA-based sealer, MTA Fillapex. The superior solubility resistance of the bioceramic sealers can be attributed to their inherent chemical stability and ability to form a stable bond with the dentinal walls [20]. Although BioRoot RCS exhibits excellent biocompatibility and sealing ability, it has slightly higher solubility due to its hydrophilic nature and the potential for gradual dissolution of its inorganic components over time [21]. The results of the current study are in line with the results of previous studies which evaluated solubility of root canal sealers [11,14-16, 22-25].

The release of calcium ions from root canal sealers is another critical factor, as it contributes to maintaining an alkaline pH within the root canal system, which is unfavorable for the growth of microorganisms. The results showed that the bioceramic sealers, Endosequence and MTA Fillapex, had significantly higher calcium ion release compared to BioRoot RCS and AH Plus. The sustained calcium ion release from the bioceramic sealers helps create an alkaline environment that can inhibit the proliferation of residual microbes, thereby reducing the risk of endodontic treatment failure. AH Plus sets and cures through an epoxy-amine condensation reaction, which does not involve the release of calcium ions. Hence AH plus lacks this inherent and unique property of other calcium based sealers. The results of the current study were in agreement with the results of the previous studies assessing calcium ion release with different

endodontic sealers [17,18]. The correlation between the solubility and calcium ion release characteristics of the tested sealers provides valuable insights. The bioceramic sealers, with their low solubility and high calcium ion release, demonstrate the ability to establish a durable and alkaline root canal environment, which is crucial for long-term success. In contrast, the resin-based sealer, AH Plus, and the MTA-based sealer, MTA Fillapex, exhibited higher solubility and lower calcium ion release, which may compromise their sealing ability and antimicrobial potential over time.

The findings of this study suggest that the bioceramic sealer such as Endosequence, possess favorable physicochemical properties that can contribute to the long-term success of endodontic treatment. The enhanced sealing ability and sustained calcium ion release from these sealers create an environment that is less conducive to microbial growth and periapical inflammation, which are common causes of endodontic treatment failure. It is important to note that the clinical performance of root canal sealers is influenced by various factors, including the quality of root canal preparation, the adaptation of the sealer to the dentinal walls, and the techniques employed during obturation. Nevertheless, the physicochemical properties evaluated in this study provide valuable guidance for clinicians in selecting an appropriate root canal sealer that can maximize the likelihood of successful long-term outcomes.

Conclusion

In conclusion, the results of this study highlight the superior solubility resistance and calcium ion release characteristics of the bioceramic sealer, Endosequence compared to the BioRoot RCS and the MTA-based sealer, MTA Fillapex. Although resin sealer, AH plus exhibits lesser solubility than other sealers, it suffers from the disadvantage that it is incapable of releasing calcium ion. These findings contribute to the growing body of evidence supporting the use of bioceramic sealers in endodontic practice, as they possess the potential to enhance the long-term success of root canal treatment.

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