

# Dissolution of a mineral trioxide aggregate sealer in endodontic solvents compared to conventional sealers

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**Abstract:** The aim of this study is to evaluate the solubility of a Mineral Trioxide Aggregate sealer (MTA-Fillapex) compared with five other sealers, calcium hydroxide (Sealapex), resin (Realseal), zinc oxide-eugenol (Tubli-Seal), and two epoxy resins (AH-26 and AH-Plus), in chloroform and eucalyptoil in static and ultrasonic environments. Samples of each sealer were prepared ( $n = 180$ ) and then divided into 12 groups that were immersed in solvents for 5 and 10 min in static and ultrasonic environments. The mean weight loss was determined, and the values were compared using Student's t-test, One-way ANOVA, and Tukey's HSD post-hoc test ( $p < 0.05$ ). In chloroform, MTA-Fillapex, AH-26, and Sealapex displayed moderate solubility with no significant difference in dissolution ( $p = 0.125$ ); however, their dissolution was significantly lower than that of AH-Plus ( $p < 0.001$ ), which was almost fully dissolved after 10 minutes. Realseal was significantly less soluble than all sealers ( $p < 0.001$ ). In eucalyptoil, MTA-Fillapex showed low solubility, as all of the sealers did, but Tubli-Seal was significantly more soluble than other sealers ( $p < 0.001$ ). Using ultrasonic activation resulted in a significantly higher dissolution rate in chloroform for all sealers except MTA-Fillapex after 10 min ( $p = 0.226$ ). In eucalyptoil, ultrasonic activation significantly increased the dissolution rate of all sealers except MTA-Fillapex after 5 and 10 min, Sealapex at 10 min, and AH-Plus at 5 min ( $p > 0.05$ ). In conclusion, MTA-Fillapex was not sufficiently dissolved in either solvent. Ultrasonic activation had limited effectiveness on MTA-Fillapex dissolution, whereas it significantly increased the efficiency of solvents in dissolving a number of endodontic sealers.

**Keywords:** Endodontics; Root Canal Filling Materials; Solubility; Solvents.

## Introduction

The goal of root canal treatment is to disinfect and clean the root canal system and to eventually seal the system and thereby prevent recontamination. A success rate of 97% has been reported for initial endodontic treatment,<sup>1</sup> but failure still occurs during post-treatment. Because persistent or reintroduced microorganisms are the major cause of post-treatment disease,<sup>2</sup> it can be assumed that the obturation material



and root canal sealer were infected. Thus, the aim is to maximize their removal to optimize debridement and bacterial control.

The most widely used root canal filling material is gutta-percha in conjunction with a root canal sealer. Currently, several root canal sealers that are based on different formulas are available. Ørstavik<sup>3</sup> categorized sealers into five different types according to their chemical formulation: zinc-oxide-eugenol based sealers, glass-ionomer based sealers, resin-based sealers, silicone-based sealers, and calcium hydroxide-based sealers. Recently, new sealers have been introduced into the market, including Bioceramic sealers and mineral trioxide aggregate-based sealers.<sup>4</sup> Furthermore, sealers based on MTA are biocompatible and induce mineralization.<sup>5</sup> They exhibit a stronger adhesiveness to dentin than do conventional zinc oxide/eugenol-based sealers,<sup>6</sup> and they possess a sealing ability comparable to that of epoxy resin-based sealers.<sup>7</sup>

Several studies have suggested that chloroform is the most efficient solvent for most root filling materials compared with eucalyptoil, halothane, and xylol.<sup>8,9,10,11,12</sup> Its safety and efficacy have also been proven over the years.<sup>13,14</sup> However, chloroform has been reported to be potentially harmful when extruded into the periapical tissue.<sup>15</sup> It can be tissue-toxic and has been shown to be potentially carcinogenic. Essential oils have been used in endodontics because of their proven safety, biocompatibility and non-carcinogenicity compared with organic solvents.<sup>13</sup> Therefore, choosing the appropriate solvent necessitates the existence of a balance between its effectiveness and safety in usage.<sup>16</sup>

In an attempt to promote the efficacy of irrigation solutions and organic solvents, several methods have been devised to augment the irrigation process. The application of mechanical agitation and the activation of the solutions have been shown to improve the disinfection of root canals.<sup>17</sup> However, there is a lack of literature evaluating the effect of ultrasonic activation on the efficiency of organic solvents during retreatment.

While root canal filling material should preferably display reasonable solubility in chemical solvents to facilitate retreatment, a high solubility of the material is undesirable because dissolution may result in

gap formation both within the material and at the dentine material interface, which predispose the tooth to bacterial leakage over time;<sup>3</sup> therefore, there are international specifications in this regard based on ANSI/ADA Specification 57.<sup>18</sup> This study is based on the fact that removing endodontic sealers from the root canal system by using solvents is a must for effective cleaning and disinfection.

Numerous studies have assessed the dissolving efficacy of organic solvents on various commonly used root canal sealers.<sup>9,11,19,20,21,22</sup> However, not enough information is available regarding the solubility properties of the MTA-based root canal sealers in common endodontic solvents. Therefore, the aim of the present study is to assess the solubility of MTA-Fillapex compared with five sealers, calcium hydroxide (Sealapex), resin (Realseal), zinc oxide-eugenol (Tubli-Seal), and two epoxy resin sealers (AH-26 and AH-Plus), using two solvents (Chloroform, Eucalyptoil) in both static and dynamic environments. The null hypothesis tested was that MTA-Fillapex is less soluble than other tested sealers in both chloroform and eucalyptoil in static and ultrasonic environments.

## Methodology

### Material selection

The solubility of six root canal sealers (Table 1) was assessed in chloroform (99% Chloroform, Carl Roth, Karlsruhe, Germany), eucalyptoil (Biodinamica Quim. e Farm. Ltda., Ibiporã, Brazil), and distilled water as a control solution.

### Sample molds design

A stainless steel ring with an outer diameter of 10 mm and a thickness of 2 mm was used. A 4-mm-diameter centrally located hole was drilled in each mold for specimen placement,<sup>23</sup> and a small hole was drilled peripheral to the sample hole to suspend the molds in the solution.

### Sealer sample preparation

Prior to placing the mixed sealer samples in the stainless steel molds, all of the molds were cleaned with acetone for 15 min and weighed before use. Then,

**Table 1.** Root canal sealers used in the study.

| Root canal sealer | Chemical group of sealer | Composition of sealer   | Manufacturer   |
|-------------------|--------------------------|---|--|
| MTA-Fillapex      | MTA                      | Salicylate resin, diluting resin, natural resin, bismuth trioxide, nanoparticulated silica, MTA, pigments   | Angelus Soluções Odontológicas, Londrina, PR, Brazil |
| Sealapex          | Calcium hydroxide based  | Base: Calcium Hydroxide, Sulfonamide, zinc oxide, zinc stearate, Colloidal silica<br>Catalyst: Polymethylene salicylate, isobutyl Salicylate, resin, Barium sulfate, Titanium oxide, Colloidal silica   | Kerr, Salerno, Italy                                 |
| Tubli-Seal        | Zinc oxide eugenol       | Base: Zinc oxide, Oleo resin, Bismuth trioxide, Thymol iodide, oils and waxes<br>Catalyst: Eugenol, Polymerized resin, Annidalin  | Kerr, Salerno, Italy                                 |
| Realseal SE       | Resin based              | Urethane dimethacrylate, polyethylene glycol dimethacrylate, ethoxylated bisphenol-A dimethacrylate, Bisphenol glycidyl dimethacrylate, barium borosilicate glass, barium sulfate, silica, calcium hydroxide, Bismuth oxychloride with amines.            | SybronEndo Corporation, Orange, USA                  |
| AH-26             | Epoxy resin based        | Powder: Bismuth oxide, Methenamine, Titanium oxide<br>Liquid: Bisphenol-A-diglycidylether   | Dentsply DeTrey GmbH, Konstanz, Germany              |
| AH-Plus           | Epoxy resin based        | Paste A: Bisphenol A epoxy resin, Bisphenol F epoxy resin, Calcium tungstate, Zirconium oxide, Silica, Iron Oxide Pigments<br>Paste B: Dibenzyl diamine, Aminoadamantane, Tricyclodecane-diamine, Calcium tungstate, Zirconium oxide, Silica, Silicon oil | Dentsply DeTrey GmbH, Konstanz, Germany              |

they were placed on a glass plate and slightly filled to excess with the freshly prepared sealer using a 1-mL syringe to prevent air entrapment. MTA-Fillapex and Realseal were supplied with a self-mixing tip, which was used for mixing the sealer and placing it into the metal rings directly from the tip. All sealers were mixed according to manufacturer's instructions. Because calcium hydroxide-based sealer requires moisture for setting; Sealapex was mixed with a spatula moistened with tap water.<sup>11</sup> Ten minutes after the sealer mixtures were first prepared, they were left to set up for at least 48 h and up to 7 d, as in the case of MTA-Fillapex and Sealapex, at 37°C and 100% humidity. The samples were considered to be completely set when a clean explorer did not penetrate the test sample. Excess material was trimmed off to the level of the mold surface using a scalpel. The samples were weighed on a digital scale to four decimal places of a gram.

## Experiment set-up

### The first part of the experiment (static environment)

At room temperature, each sealer sample was immersed in 20 mL of each solvent in a plastic tube with a screw cap (Chloroform, Eucalyptoil and distilled water as control group) for 5 and

10 min (n = 15 for each immersion period). After the specified immersion period, the sealer samples were removed from the solvents and allowed to dry for 24 h at 37°C before their weights were measured. Twenty-five empty sample molds were immersed in chloroform or in eucalyptoil for 5 and 10 min to ensure that the molds' suspension in solvents did not affect their weight.<sup>9</sup>

### The second part of the experiment (ultrasonic activation)

Sample preparation was carried out as in the first part of the experiment (n = 15 for each immersion period), where each sealer sample was immersed in 20 mL of each solvent as mentioned previously. The plastic tubes containing the metal rings with the set sealer were subsequently placed in an ultrasonic chamber. This was done by placing the plastic tubes alternately in the ultrasonic chamber for one minute and then in a static environment during the second minute, and so on, throughout the designated immersion periods. The plastic tubes were placed in a Rack so they would not touch the floor of the ultrasonic chamber. At the end of each immersion period, the samples were removed and left to dry for 24 h at 37°C.

Following the drying period, all samples were weighed three times, and the weight of the sealer

was recorded to the nearest 0.0001 gramme. The amount of sealer removed from the specimen was determined as the difference between the original weight of the sealer and its final weight and measured to the nearest 0.0001 gramme. This difference in weight was calculated as a percentage of the original weight, recorded to the nearest 0.001%.<sup>9</sup>

The collected data were analyzed statistically using Statistical Software (SPSS for Windows, Release 6.1.2, IBM SPSS, Armonk, USA). Parametric tests were performed because the assumption of data being normally distributed was confirmed using the Leven's test. Independent sample t-test was used for pairwise comparisons, and one-way ANOVA with Tukey's HSD post-hoc test ( $p < 0.05$ ) was used for multiple comparisons.

## Results

The mean values and standard deviation of weight loss percent of sealers are shown in Table 2. No change in the weight of empty molds was reported in different immersion periods or environments in either solvent. In the distilled water control group, no sealer was dissolved in a static environment; however, in an ultrasonic environment, MTA-Fillapex and Sealapex showed significantly higher solubility after 10 min, but their dissolution values in distilled water were low compared with those of organic solvents (0.143% and 0.162%, respectively;  $p < 0.001$ ).

Chloroform was a far more effective solvent for all root canal sealers compared with eucalyptoil in both static and ultrasonic environments (Figure). In addition, the solubility of sealers significantly increased as the immersion period increased from 5 to 10 min, except for Realseal in chloroform in a static environment (Figure). The solubility of MTA-Fillapex in chloroform was comparable to that of Sealapex and AH-26 but was significantly lower than AH-Plus and Tubli-Seal ( $p < 0.001$ ). Furthermore, the solubility of AH-Plus was significantly higher than that of all other sealers in chloroform in both the static and ultrasonic environments ( $p < 0.001$ ) (Figure).

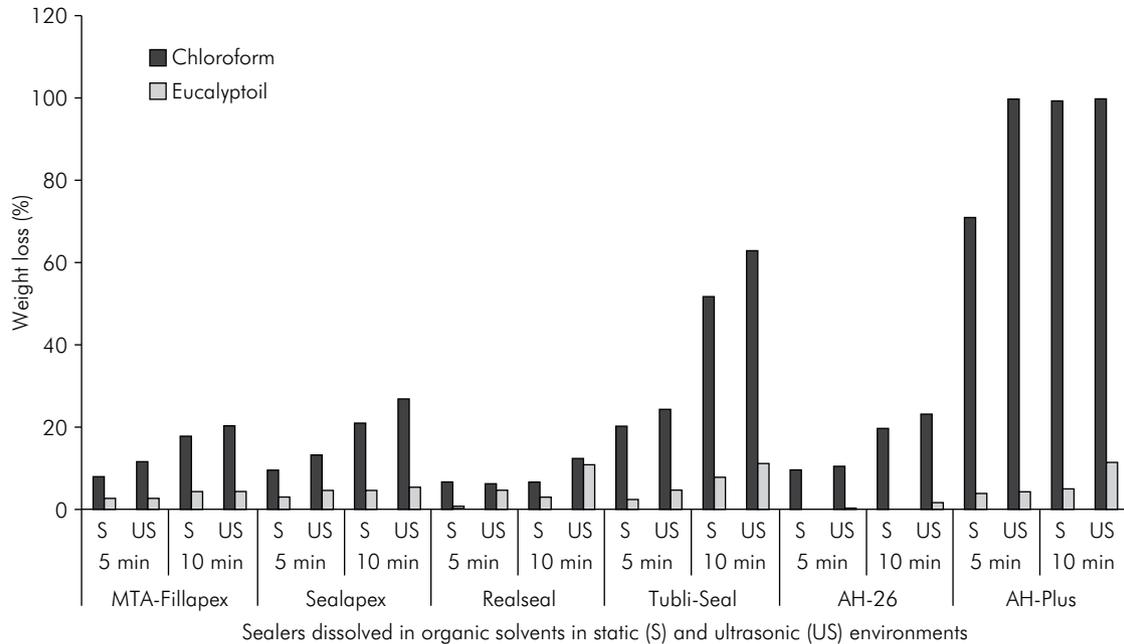
Generally, the efficiency of eucalyptoil in dissolving the various sealers was poor in both environments compared with chloroform, as shown in Figure 1. The solubility of MTA-Fillapex, AH-Plus, and Sealapex after 10 min was significantly higher than that of Realseal and AH-26 ( $p < 0.05$ ). Tubli-Seal was significantly more soluble than other sealers in eucalyptoil at 10 min of immersion, but it still showed a low solubility range. In contrast, AH-26 was virtually insoluble with less than 1% weight loss after 10 min of immersion.

## Effect of ultrasonic activation on sealer dissolution

In chloroform, ultrasonic activation during sealer immersion significantly increased sealer dissolution after 5 min, except for Realseal and AH-26 ( $p = 0.819$ ,

**Table 2.** Mean values and standard deviation (SD) of sealer dissolution in Distilled water, Chloroform, and Eucalyptoil in static and ultrasonic environments.

| Root canal sealer | Time   | Distilled water         |                             | Chloroform              |                             | Eucalyptoil             |                             |
|-------------------|--------|-------------------------|-----------------------------|-------------------------|-----------------------------|-------------------------|-----------------------------|
|                   |        | Static (%)<br>Mean (SD) | Ultrasonic (%)<br>Mean (SD) | Static (%)<br>Mean (SD) | Ultrasonic (%)<br>Mean (SD) | Static (%)<br>Mean (SD) | Ultrasonic (%)<br>Mean (SD) |
| MTA-Fillapex      | 5 min  | 0.0000 (0.0000)         | 0.0000 (0.0000)             | 8.2521 (1.7175)         | 11.9293 (1.9527)            | 3.0001 (0.7763)         | 2.9381 (1.1206)             |
|                   | 10 min | 0.0000 (0.0000)         | 0.1431 (0.0003)             | 18.0221 (2.6439)        | 20.4713 (3.2557)            | 4.2666 (1.1239)         | 4.3965 (1.7271)             |
| Sealapex          | 5 min  | 0.0000 (0.0000)         | 0.0000 (0.0000)             | 9.6648 (2.6796)         | 13.5091 (1.8419)            | 3.2717 (0.9489)         | 4.7707 (2.2573)             |
|                   | 10 min | 0.0000 (0.0000)         | 0.1622 (0.0009)             | 21.1981 (3.3247)        | 27.0490 (4.1278)            | 4.8847 (1.4484)         | 5.7582 (1.5581)             |
| Realseal          | 5 min  | 0.0000 (0.0000)         | 0.0000 (0.0000)             | 6.8663 (2.2195)         | 6.7082 (1.4450)             | 1.3310 (0.5559)         | 4.8431 (1.7197)             |
|                   | 10 min | 0.0000 (0.0000)         | 0.0000 (0.0000)             | 6.8445 (2.3839)         | 12.4938 (1.5978)            | 3.3424 (1.0234)         | 11.0623 (2.2372)            |
| Tubli-Seal        | 5 min  | 0.0000 (0.0000)         | 0.0000 (0.0000)             | 20.5879 (4.1845)        | 24.5932 (4.8071)            | 2.8478 (0.7118)         | 4.9254 (1.1626)             |
|                   | 10 min | 0.0000 (0.0000)         | 0.0000 (0.0000)             | 52.0731 (8.3568)        | 63.3796 (14.6160)           | 8.1419 (1.7268)         | 11.6392 (6.2900)            |
| AH-26             | 5 min  | 0.0000 (0.0000)         | 0.0000 (0.0000)             | 9.6467 (1.8443)         | 10.6571 (2.6931)            | 0.1665 (0.0313)         | 0.6912 (0.3226)             |
|                   | 10 min | 0.0000 (0.0000)         | 0.0000 (0.0000)             | 19.5736 (3.3206)        | 23.2361 (2.9783)            | 0.3507 (0.1464)         | 1.9240 (0.7772)             |
| AH-Plus           | 5 min  | 0.0000 (0.0000)         | 0.0000 (0.0000)             | 71.0912 (16.6769)       | 99.5987 (0.4258)            | 4.0956 (1.3080)         | 4.4350 (2.1001)             |
|                   | 10 min | 0.0000 (0.0000)         | 0.0000 (0.0000)             | 99.3285 (0.6671)        | 99.8871 (0.1349)            | 5.3204 (1.7283)         | 11.6647 (2.5589)            |



**Figure.** Dissolution of sealers in chloroform and eucalyptoil in static and ultrasonic environments for 5- and 10-min immersion periods.

0.241, respectively). However after 10 min, ultrasonic activation did not affect the dissolution of MTA-Fillapex but resulted in a significantly higher dissolution rate for all other tested sealers ( $p < 0.05$ ) (Figure).

In eucalyptoil, MTA-Fillapex did not show a statistically significant difference when it was dissolved in an ultrasonic environment compared with a static environment in both immersion periods (Figure). However, the remaining sealers reported significantly greater dissolution after ultrasonication, except for Sealapex at 10 min and AH-Plus at 5 minutes.

## Discussion

The null hypothesis that was postulated in the current study was rejected. All of the tested sealers in the current study showed significantly higher dissolution in chloroform than in eucalyptoil. In chloroform, MTA-Fillapex showed a lower dissolution rate than did AH-Plus and Tubli-Seal but a similar dissolution rate to AH-26 and Sealapex. Similar results were seen when ultrasonic activation was used.

The method used in the current study to assess sealer dissolution was adopted due to its simplicity and reproducibility and fairly low cost. However, several clinically relevant parameters, such as root

canal dimensions, solvent temperature, and contact time, could not be replicated. It can be assumed that the contact area in the current study between the sealer and solvent solution was higher than the actual contact area with the sealer within the confines of the root canal anatomy, which may imply a lower dissolution in a clinical setting. Nonetheless, it facilitated the comparison of the results with previously published data.<sup>9,11,21,22,23</sup>

In the distilled water control group and in an ultrasonic environment, MTA-Fillapex and Sealapex showed a significantly higher degree of solubility after 10 min of immersion, which may have occurred because both sealers release  $\text{Ca}^{+2}$  ions when dissolved in water. This was described in previous studies that investigated the ion release of MTA-Fillapex and Sealapex.<sup>24,25,26</sup>

Borges et al.<sup>26</sup> reported that the solubility of MTA-Fillapex in deionized water was high (14.89%) and comparable to that of Sealapex, which is higher than the ANSI/ADA<sup>18</sup> requirements of 3% of the total mass. This solubility is higher than the value reported in the current study; the difference is due to the different experimental method and sample dimensions employed. Borges et al.<sup>26</sup> stored the samples in water for 7 days before weighing to test the

degree of dissolution of sealers, which is considered a negative point in terms of the long-term apical and coronal seal that is crucial for the long-term success of root canal therapy. In contrast, this study aimed to evaluate the dissolution of sealers in chemical solvents as part of the endodontic retreatment procedure, for which maximum solubility is required to optimize disinfection of the canals. Therefore, the samples were kept in water for 5 or 10 min only to simulate clinical procedures. The dissolution over this short storage period was thus not comparable to that observed for 7 days of storage, as reported by Borges et al.<sup>26</sup>

Overall, the results concerning the solubility of the sealers tested in the current study in chloroform and eucalyptoil are in good agreement with previously published studies.<sup>9,11,19</sup> According to our results, chloroform was a far more effective solvent than eucalyptoil for MTA-Fillapex and all of the other tested sealers. In a static environment, the solubility of MTA-Fillapex in chloroform was similar to that of Sealapex and AH-26 but significantly lower than that of AH-Plus and Tubli-Seal. This could be explained by the resinous component in the MTA-Fillapex.<sup>4</sup>

AH-Plus and Tubli-Seal were highly soluble in chloroform at an exposure time of 10 min, which is in agreement with other studies.<sup>9,19,21</sup> AH-26 was found to be significantly less soluble than AH-Plus. This finding agrees with the finding of Hansen,<sup>27</sup> who reported that chloroform did not have a noticeable dissolving effect on AH-26; similarly, Schäfer and Zandbiglari<sup>9</sup> found a great difference in the dissolution rate between AH-26 and AH-Plus. This discrepancy might be related to the difference in the size of their resinous particles. It could also be due to differences in their manufacturing details, which most manufacturers do not disclose.

Epiphany sealer, which has a similar chemical composition to Realseal but a different brand name, has been reported to have a good solubility in chloroform after a 10-min immersion.<sup>19</sup> However, this disagrees with our results concerning Realseal. This discrepancy might be due to the difference in methodologies used. Furthermore, Bordumulu et al.<sup>19</sup> removed the sealer samples from the molds and then immersed them in the solvent, which provided a three-dimensional

exposure of the sealer to the solvents. However, in this present study, only 2 surfaces of the sealer were exposed to the solvent.

Only Tubli-Seal was partially dissolved by eucalyptoil. This might have occurred because most ZnOE-based cements have a high concentration of colophony in their composition to improve their adhesive properties. Colophony is a vegetable resin that is composed of approximately 90% of resinous acids and is soluble in solvents that are commonly used in endodontic retreatment.<sup>22</sup>

No previous studies have investigated the effect of ultrasonic activation on the dissolution of root canal sealers. In the current study, the plastic tubes containing the metal rings with the set sealer were placed in an ultrasonic chamber. The agitation effect of the ultrasonic activation was noted by the vibrations seen in the solvent solution surrounding the sealer samples in the plastic tubes. In the root canal setting, the bond strength of the sealer to dentin may further affect the degree of dissolution by ultrasonic agitation; however, the methodology is acceptable for the sake of comparison between different sealers.

Applying ultrasonic activation converts electric energy into waves with certain frequencies. This energy generates a high movement of fluid in a circular action around the vibrating instrument. This rapid movement occurs inside the canal space when activating an irrigant and should induce its cleaning and dissolving efficiency. In addition, ultrasonically activating a solution can cause cavitation (*i.e.*, the development and collapse of bubbles in a liquid medium followed by the release of energy) of the irrigant, which also improves its efficiency.<sup>28</sup> This explains our results, which showed a significantly higher efficiency of the tested solvents in dissolving root canal sealers when using ultrasonic activation.

Generally, immersing sealers in organic solvents for 10 min resulted in a significantly increased dissolution of sealers in both static and ultrasonic environments compared with 5 min, which is in agreement with previous studies.<sup>9,11,19,21</sup> Schäfer and Zandbiglari<sup>9</sup> reported a remarkable increase in sealer solubility in chloroform and eucalyptoil with time, especially after a 5-min immersion period. That result excludes

AH-Plus and AH-26 in eucalyptoil, which showed very low solubility at all immersion periods. Similarly, Bordumulu et al.<sup>19</sup> reported no significant difference between 2 and 5 min of immersion in chloroform and eucalyptoil for AH-Plus, Ketac-Endo and Epiphany, but the 10-min immersion period showed a significantly higher dissolution rate compared with 2 and 5 min immersion in either solvent. In contrast, Martos et al.<sup>22</sup> found no statistically significant difference in the dissolution of 3 different sealers (Sealer 26, Endofill and RoekoSeal) when they were immersed for 2 and 10 min in eucalyptoil.

The results of this study showed that the use of an ultrasonic activation or the dissolution of MTA-Fillapex in organic solvents for longer periods of time might aid in obtaining a higher dissolution rate for this sealer. Compared with the other sealers that were tested in the current study, its dissolution is comparable to that of AH-26 but is still considered low compared with commonly used sealers such as AH-Plus. Therefore,

this finding should be considered when attempting to use MTA-Fillapex sealer in clinical cases.

## Conclusions

Within the limitations of this study, the following conclusions can be drawn:

- a. Chloroform presented a superior solvent effect compared with eucalyptoil, with a significant dissolution of tested sealers.
- b. Realseal displayed the lowest solubility in chloroform, whereas in eucalyptoil, AH-26 was the least soluble of the tested sealers.
- c. Generally, ultrasonic activation significantly increased the efficiency of chloroform but not eucalyptoil in dissolving sealers.

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