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Effect of Formic Acid and Passive Ultrasonic Irrigation on the Removal of Residual Filling Material Containing AH-Plus Bioceramic Sealer

Shirley Granados-Laura [a*], Carlos Liñan-Duran [a], Carmen Garcia-Rupaya [a]

a School of Dentistry, Universidad Peruana Cayetano Heredia, Lima, Peru

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**Corresponding author*: Shirley Granados-Laura, DDS, MSc. School of Dentistry, Universidad Peruana Cayetano Heredia, Av. Salaverry 2475, Lima 00051, Peru

Email: shirley.granados@upch.pe

Abstract

Introduction: Root canal retreatment is challenged by the strong adhesion of sealers to dentin. AH-Plus Bioceramic, an epoxy resin-based sealer containing calcium silicate components, could present particular difficulty during removal. This study evaluated whether 5% formic acid with passive ultrasonic activation (PUI) is as effective as 10% formic acid and 2% sodium hypochlorite (NaOCl) in the removal of residual filling material of teeth obturated with AH-Plus Bioceramic sealer. **Materials and Methods:** Thirty-three lower premolar roots were obturated with AH-Plus Bioceramic sealer using a single-cone technique. After instrumentation with the Reciproc R40 file, the roots were randomly assigned to three groups of final irrigation: 5% formic acid, 10% formic acid, and 2% NaOCl, all combined with PUI. The volume of filling material was measured using cone-beam computed tomography (CBCT) analysis at three stages: after obturation, after instrumentation for retreatment, and after final irrigation with formic acid or NaOCl combined with PUI. Statistical analysis included Friedman and Wilcoxon tests for intragroup comparisons and the Kruskal–Wallis test with Dunn's post hoc test for intergroup comparisons. **Results:** All irrigants combined with PUI reduced the filling material volume with no significant differences among them (P>0.05). **Conclusion:** Ultrasonically activated 5%, 10% formic acid, and 2% NaOCl showed similar removal of residual filling material containing AH-Plus Bioceramic sealer.

Keywords: Cone-beam Computed Tomography; Formic Acid; Root Canal Retreatment; Silicate Cement; Ultrasonics

Introduction

Non-surgical root canal retreatment aims to remove filling materials and microorganisms from previously treated canals using mechanical and chemical techniques [1]. However, complete removal remains difficult, and treatment outcomes depend on factors such as the type of sealer used [2, 3].

Calcium silicate-based sealers bond to dentin through hydroxyapatite formation and penetrate dentinal tubules [4, 5]. When used with the single-cone technique, they result in greater sealer volume, which complicates removal [6]. AH-Plus Bioceramic (Dentsply Sirona, Charlotte, NC, USA), an epoxy resin–based sealer containing calcium silicate components [7], exhibits some of these characteristics, although it differs from conventional hydraulic calcium silicate sealers.

Formic acid has been proposed as a solvent for retreatment procedures in teeth obturated with calcium silicate-based sealers. Garrib and Camilleri [6] demonstrated that 10% formic acid facilitates removal because this concentration promotes dentin demineralization [8]. At higher concentrations in the construction industry, it dissolves Portland cement, which has a chemical composition similar to that of calcium silicate-based sealers [6, 9]. In vitro studies have reported removal rates of up to 95% [6], with minimal adverse effects on dentin [1, 6]. Although high concentrations of formic acid are cytotoxic [10, 11], lower concentrations may provide effective dissolution with improved safety.

Passive ultrasonic irrigation (PUI) is the agitation of irrigant solutions within the root canal system, wherein a small file or smooth wire oscillates freely to induce powerful acoustic microstreaming [12]. In the context of retreatments, PUI increases the amount of filling material removed by generating acoustic transmission within the solutions, which enhances sealer detachment, particularly in areas inaccessible to instruments [13, 14]. No studies have evaluated lower concentrations of formic acid in association with PUI.

Therefore, this study aimed to assess whether 5% formic acid combined with PUI is as effective as 10% formic acid and 2% NaOCl in removing filling material from teeth obturated with AH-Plus Bioceramic sealer, using cone-beam computed tomography (CBCT) for volumetric assessment. The null hypothesis was that the PUI of 5% formic acid activation is as effective as 10% formic acid and 2% NaOCl in reducing residual filling material.

Materials and Methods

Ethical approval was obtained from the Institutional Research Ethics Committee of Universidad Peruana Cayetano Heredia (CIEI-464-43-23). The sample size was determined from pilot data using Epidat 4.2 (Health Department, Xunta de Galicia, Galicia, Spain), considering a mean difference of 1.905, α=0.05, and a statistical power of 0.80. A total of 33 single-rooted mandibular premolars extracted for orthodontic purposes were included. Teeth presenting curvature greater than 10 [15], calcifications, internal or external resorption, incomplete root development, or previous endodontic treatment were excluded. Radiographs in both projections confirmed eligibility. All interventions were performed by the same operator (SGL).

The teeth were decoronated to a standardized root length of 14 mm. Following access preparation, canal patency was achieved, and the working length was established 1 mm short of the apical foramen. Instrumentation was performed to the working length using a Reciproc R40 file (VDW, Munich, Germany) under irrigation with 5 mL of 2% NaOCl. Final irrigation included 6 mL of 2% NaOCl for 1 min, 1 mL of 17% EDTA for 3 min, and 5 mL of saline solution for 3 min, all delivered through a 30G needle positioned 1 mm short of the working length [6].

The canals were dried with paper points and obturated using a single-cone technique with an R40 gutta-percha cone (VDW, Munich, Germany) and AH-Plus Bioceramic sealer (Dentsply Sirona, Charlotte, USA) to the working length. The canal access was restored with Filtek Z350 XT resin (3M ESPE, Minnesota, USA). Specimens were then stored at 37°C and 100% humidity in an incubator for one week.

After obturation, the resin restoration was removed. The cervical one-third of the gutta-percha was removed using a 40/0.04 heat-activated plugger (Fast Pack, Eighteeth, Changzhou, China), and the remainder was removed with a Reciproc R40 file under irrigation with 2% NaOCl until the file reached the working length. Apical patency was verified with a #15 K-file, and canals were dried with paper points.

Then, the specimens were randomly distributed into three groups (n=11):

1. 5% formic acid with PUI
2. 10% formic acid with PUI
3. 2% NaOCl with PUI (control group)

The PUI was performed with the gold tip (0.20, 2% taper, 18 mm) of the Ultra X ultrasonic device (Eighteeth, Changzhou, China), positioned 2 mm short of the working length and operated at a frequency of 45 kHz in high-output power mode [16]. The tip was passively introduced into the canal and kept centered with gentle pendulum movements toward the polar areas, following the protocol described by Crozeta *et al.* [13], to standardize the irrigation procedure and ensure reproducibility of the experimental conditions. Irrigants (formic acid and NaOCl) were applied at 0.3 mL for 2 min [1], followed by ultrasonic activation in three cycles of 20 sec each according to the protocol of Van Der Sluis *et al.* [17]. Final irrigation consisted of 3 mL of 2% NaOCl for 1 min and 3 mL of saline solution for 1 min [6].

The CBCT assessment was performed at three stages: after obturation (for initial volume), after instrumentation for retreatment, and after final irrigation with formic acid or NaOCl combined with PUI (for final volume). CBCT scans were obtained using a Planmeca Promax 3D CBCT Scanner (Planmeca, Helsinki, Finland). Acquisition parameters included an isotropic voxel size of 75 µm, a copper filter with 2.5 mm aluminum, 90 kV, 6.3 mA, 15 sec exposure, 5 cm FOV, and 360° rotation. Images were exported in DICOM format at a voxel size of 0.15 mm (150 µm) and analyzed using ITK-SNAP 4.0 software (Penn Image Computing and Science Laboratory, Penn, USA) [18]. The "Snake" semi-automatic segmentation tool was applied to select hyperdense regions corresponding to obturation material. The "Thresholding" mode was then set at a minimum intensity of 2600 HU to identify hyperdense canal contents. This threshold was established during a pilot calibration study in consultation with an oral and maxillofacial radiologist and was subsequently applied to all specimens to ensure consistency in volumetric measurements. The "Bubble" tool was used to quantify obturation material volume in mm^3.

Intraobserver reliability was assessed with the intraclass correlation coefficient (ICC=0.9). All measurements were performed by a single blinded evaluator (SGL).

Statistical analysis

Data were analyzed using Stata 18 (StataCorp, College Station, TX, USA). Normality was assessed with the Shapiro–Wilk test, which indicated a non-normal distribution. Consequently, the Friedman test was applied to compare filling material volume across the three stages within each group, with pairwise comparisons performed using the Wilcoxon signed-rank test. Intergroup comparisons at each stage were conducted with the Kruskal–Wallis test, followed by Dunn's post hoc test. Statistical significance was set at 5%.

Results

There was no significant difference among the use of 5% or 10% formic acid and 2% NaOCl with PUI in the removal of filling material containing AH-Plus Bioceramic Sealer (P>0.05). Additionally, a significant reduction in the volume of residual filling material was observed when comparing initial and final measurements for all groups (P<0.05) (Table 1).

Discussion

This study compared the removal of residual filling material using 5% and 10% formic acid, or 2% NaOCl, all with PUI. The null hypothesis was accepted since no significant differences were observed among the groups. This finding suggests that a lower concentration of formic acid may represent a safer and more conservative alternative.

The clinical relevance of the present study lies in the effects on dentin rather than systemic toxicity. Acidic solutions can alter dentin microhardness and permeability, thereby facilitating tubule cleaning and sealer filling material removal [1]. Garrib and Camilleri [6] previously reported that 10% formic acid does not damage dentin, while Rezaei *et al.* [1] demonstrated its effectiveness in opening and cleaning dentinal tubules, leaving minimal debris. At 5%, formic acid may provide similar efficacy with reduced potential adverse effects. However, further studies are needed to investigate dentin microhardness, collagen degradation, and cytotoxicity after exposure to 5% formic acid. Previous studies have suggested that formic acid may interact with calcium-containing materials, producing soluble salts such as calcium formate. The reaction of formic acid with calcium compounds produces soluble salts such as calcium formate [6, 9, 19], which could contribute to the removal of calcium silicate-based sealers.

In addition, PUI may enhance irrigant penetration and disruption of filling material through cavitation and acoustic streaming may enhance this effect through cavitation and acoustic streaming, disrupting sealer structure and improving irrigant penetration [13].

However, because the present methodology quantified total residual filling material volume and did not differentiate between gutta-percha and sealer remnants, these mechanisms should be regarded as potential explanations rather than direct evidence derived from the present study.

The comparable removal achieved with 5% and 10% formic acid suggests that both concentrations may be effective when combined with PUI. Although the observed results may be related to interactions between formic acid and calcium silicate-containing materials, as well as the mechanical energy generated by ultrasound, the present study was not designed to distinguish the relative contribution of gutta-percha and sealer removal. Therefore, any material-specific interpretation should be made with caution. AH-Plus Bioceramic contains a lower proportion of tricalcium silicate (5%–15% tricalcium silicate) [7, 23], exhibits higher solubility [20, 21], and lower bond strength [22] than conventional hydraulic calcium silicate-based sealers. These properties may have influenced the observed removal pattern, although this could not be directly verified in the present study.

Table 1. Mean volume and standard deviation (mm³) of filling material at three stages (after obturation, after instrumentation, and after final irrigation with PUI), according to experimental group

Group	After obturation	After instrumentation	After final irrigation with PUI	Intragroup P-value
5% Formic acid	27.28 (7.85) ^{A}	1.54 (1.59)^{AB}	0.39 (0.63)^{B}	<0.001*
10% Formic acid	24.78 (7.23) ^{A}	1.92 (1.62)^{AB}	0.89 (1.43)^{B}	<0.001*
2% NaOCl	24.29 (9)^{A}	0.96 (1.57)^{AB}	0.14 (0.15)^{B}	<0.001*
Intergroup P-value	0.601	0.093	0.940	

Different superscript letters within the same row indicate statistically significant differences among stages within each group (Friedman test with Wilcoxon signed-rank post hoc test, P<0.05). Intergroup comparisons at each stage were performed using the Kruskal–Wallis test followed by Dunn's post hoc test (P<0.05)

The use of PUI was associated with increased filling material removal compared with mechanical instrumentation alone [13, 14]. Previous studies confirm that ultrasound enhances irrigant action, raises solution temperature, and facilitates detachment of obturation material [13, 24]. Although statistically significant intragroup reductions were observed, the residual volumes were small. Therefore, the clinical relevance of these differences should be interpreted with caution, as statistical significance does not necessarily imply clinical significance.

The present study did not use a control group using distilled water or saline, which represents a limitation. Future studies should include a saline control group to isolate the independent effect of PUI and assess applicability to sealers with higher calcium silicate content. However; it is well-known that the mechanical effect of ultrasonic tips cannot be separated from the chemical action of the irrigants. In addition, given that this was an *in vitro* study, the findings may not be generalizable to sealers with higher calcium silicate content. These findings should be interpreted with caution.

Another limitation of the present study is that CBCT image analysis was performed using datasets exported at a voxel size of 150 µm, despite image acquisition at 75 µm. Although this approach standardized image processing and segmentation across all specimens, it may have reduced the detection sensitivity for very small residual filling material volumes. In addition, CBCT-based volumetric measurements are susceptible to beam-hardening artifacts, partial-volume effects, and threshold-selection bias [25]. To minimize variability, a threshold value established during a pilot calibration study was applied consistently to all specimens. Nevertheless, the reported residual volumes, particularly the smallest measurements, should be interpreted with caution. Therefore, although significant differences were observed among the experimental groups, the accuracy of very small residual volume measurements may have been affected by the inherent limitations of CBCT image segmentation. Future studies using higher-resolution imaging techniques, such as micro-computed tomography, are warranted to confirm these findings.

Conclusions

The use of 5% formic acid with PUI demonstrated removal of filling material containing AH-Plus Bioceramic Sealer comparable to that achieved with 10% formic acid and 2% sodium hypochlorite. Future studies should include a saline control group to isolate the independent effect of ultrasonic activation and assess applicability to sealers with higher calcium silicate content.

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Conflict of interest

The authors declare no conflict of interest.

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Author contributions

Conceptualization: SLS/CGR, Methodology: SGL/CGRC/CLD; Formal analysis and investigation: SGL, Writing original draft: SGL; Writing review and editing: SGL/CGR/CLD; Supervision: CGR/CLD. All authors read and approved the final manuscript.

References

1. Rezaei G, Liu X, Jalali P. Efficacy of different solvents for achieving patency in teeth obturated using bioceramic sealer. J Endod. 2023;49[2]:219–23.
2. Yang X, Lan J, Ji M, Tsauo C, Gao Y, Zou L. Assessment of the effectiveness of supplementary methods for residual filling material removal using micro-computed tomography: A systematic review and meta-analysis of in vitro studies. Eur Endod J. 2022;7[3]:178–86.
3. Uzunoglu E, Yilmaz Z, Sungur DD, Altundasar E. Retreatability of root canals obturated using gutta-percha with bioceramic, MTA and resin-based sealers. Iran Endod J. 2015;10[2]:93–8.
4. Jeong JW, DeGraft-Johnson A, Dorn SO, Di Fiore PM. Dentinal Tubule Penetration of a Calcium Silicate–based Root Canal Sealer with Different Obturation Methods. J Endod. 2017;43[4]:633–7.
5. Salles LP, Gomes-Cornélio AL, Guimarães FC, Herrera BS, Bao SN, Rossa-Junior C, et al. Mineral trioxide aggregate-based endodontic sealer stimulates hydroxyapatite nucleation in human osteoblast-like cell culture. J Endod. 2012;38[7]:971–6.
6. Garrib M, Camilleri J. Retreatment efficacy of hydraulic calcium silicate sealers used in single cone obturation. J Dent. 2020;98:103370.
7. Sanz JL, López-García S, Rodríguez-Lozano FJ, Melo M, Lozano A, Llena C, et al. Cytocompatibility and bioactive potential of AH Plus Bioceramic Sealer: An in vitro study. Int Endod J. 2022;55[10]:1066–80.

8. Khangura AK, Gupta S, Gulati A, Singh S. Tooth decalcification using different decalcifying agents - A comparative study. J Oral Maxillofac Pathol. 2021;25[3]:463–9.
9. Pavlik V. Corrosion of hardened cement paste by acetic and nitric acids Part I: Calculation of corrosion depth. Cem Conc Res. 1994;24[3]:551–62.
10. Zhu BT. Biochemical mechanism underlying the pathogenesis of diabetic retinopathy and other diabetic complications in humans: the methanol-formaldehyde-formic acid hypothesis. Acta Biochim Biophys Sin [Shanghai]. 2022;54[4]:415–51.
11. Johnson W, Heldreth B, Bergfeld WF, Belsito D V., Hill RA, Klaassen CD, et al. Safety assessment of formic acid and sodium formate as used in cosmetics. Int J Toxicol. 2016;35[2]:41S-54S.
12. Van Der Sluis LWM, Versluis M, Wu MK, Wesselink PR. Passive ultrasonic irrigation of the root canal: A review of the literature. Int Endod J. 2007;40[6]:415–26.
13. Crozeta BM, Lopes FC, Menezes Silva R, Silva-Sousa YTC, Moretti LF, Sousa-Neto MD. Retreatability of BC Sealer and AH Plus root canal sealers using new supplementary instrumentation protocol during non-surgical endodontic retreatment. Clin Oral Investig. 2021;25[3]:891–9.
14. Madarati AA, Sammani AMN, Alnazzawi AA, Alrahlah A. Efficiency of the new reciprocating and rotary systems with or without ultrasonics in removing root-canals filling with calcium silicate-based sealer [MTA]. BMC Oral Health. 2023;23[1]:1–10.
15. Schneider SW. A comparison of canal preparations in straight and curved root canals. Oral Surgery, Oral Med Oral Pathol. 1971;32[2]:271–5.
16. Adl A, Razavian A, Eskandari F. The efficacy of EndoActivator, passive ultrasonic irrigation, and Ultra X in removing calcium hydroxide from root canals: an in-vitro study. BMC Oral Health. 2022;22[1]:1–7.
17. van der Sluis LW, Vogels MP, Verhaagen B, Macedo R, Wesselink PR. Study on the influence of refreshment/activation cycles and irrigants on mechanical cleaning efficiency during ultrasonic activation of the irrigant. J Endod. 2010;36[4]:737–40.
18. Yushkevich PA, Piven J, Hazlett HC, Smith RG, Ho S, Gee JC, et al. User-guided 3D active contour segmentation of anatomical structures: Significantly improved efficiency and reliability. Neuroimage. 2006;31[3]:1116–28.
19. Allahverdi A, Škvára F. Acidic corrosion of hydrated cement based materials part 2. - Kinetics of the phenomenon and mathematical models. Ceram - Silikaty. 2000;44[4]:152–60.
20. Quaresma SAL, Alves dos Santos GN, Silva-Sousa AC, Camargo RV, Lopes-Olhê FC, Silva-Sousa YTC, et al. Physicochemical properties of calcium silicate cement based endodontic sealers. J Mech Behav Biomed Mater. 2024;151:106400.
21. Raman V, Camilleri J. Characterization and assessment of physical properties of 3 single syringe hydraulic cement–based sealers. J Endod. 2024;50[3]:381–8.
22. Stiklaru I, Lalum E, Hamoud S, Paz M, Levin A, Ben Itzhak J, et al. The ability of different compositions of calcium silicate and epoxy sealers to withstand gutta percha removal via in vitro pull-out testing. BDJ Open. 2024;10[1]:1–5.
23. Loyola-Cano MJ, García-Rupaya CR. Evaluation of the Radiopacity, pH, and Calcium Ion Release of Calcium Silicate and Epoxy Resin-based Endodontic Cements. Iran Endod J. 2025;20[1]:1–8.
24. Macedo RG, Verhaagen B, Fernandez Rivas D, Gardeniers JGE, Van Der Sluis LWM, Wesselink PR, et al. Sonochemical and high-speed optical characterization of cavitation generated by an ultrasonically oscillating dental file in root canal models. Ultrason Sonochem. 2014;21[1]:324–35.
25. Rodrigues CT, Jacobs R, Vasconcelos KF, Lambrechts P, Fisher Rubira-Bullen IR, Gaêta-Araujo H, et al. Influence of CBCT-based volumetric distortion and beam hardening artefacts on the assessment of root canal filling quality in isthmus-containing molars. Dentomaxillofacial Radiol. 2021;50[5]:20200503.

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