

Original Research**Comparative Evaluation of Three Different Solvents in Removal of Gutta Percha Sealed with Bioceramic Sealer – An In Vitro Study**Sadhvi Pateriya ¹, Anu Narang ², Santosh Kumar Singh ³, Shivani Rawat ⁴, Gayatri H. Giramkar ⁵,
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ABSTRACT

Introduction : Successful endodontic retreatment depends on the complete removal of existing obturating material to allow effective cleaning, disinfection, and re-obturation of the root canal system. Bioceramic sealers are widely used because of their excellent sealing ability, dimensional stability, and biocompatibility. However, their strong adhesion to dentinal walls makes retreatment challenging. Mechanical instrumentation alone is often insufficient for complete removal, thereby necessitating the use of chemical solvents. Weak organic acids such as acetic acid, citric acid, and maleic acid have shown potential in dissolving calcium-based bioceramic sealers.

Aim and Objective : The aim of the study was to comparatively evaluate the efficacy of 2% acetic acid, 10% citric acid, and 7% maleic acid in the removal of gutta-percha obturated with a bioceramic sealer during endodontic retreatment.

Materials and Methods: Forty-five extracted human single-rooted teeth were selected for the study. Biomechanical preparation was completed using ProTaper Universal rotary files up to F3, followed by obturation with gutta-percha and bioceramic sealer. The samples were randomly divided into three experimental groups based on the solvent used during retreatment: Group I – 2% Acetic acid, Group II – 10% Citric acid, Group III – 7% Maleic acid. Retreatment was performed using ProTaper retreatment files (D1, D2, D3) along with the respective solvents. The teeth were longitudinally sectioned and examined under a stereomicroscope to evaluate the amount of residual obturating material in the coronal, middle, and apical thirds.

Results : None of the solvents achieved complete removal of obturating material. Residual material was observed in all specimens, with maximum remnants present in the apical third. Among the solvents tested, 7% maleic acid showed the least residual obturating material, indicating the highest efficacy, followed by 10% citric acid and 2% acetic acid. The differences among the groups were statistically significant ($p < 0.05$).

Conclusion: Within the limitations of this in-vitro study, 7% maleic acid demonstrated superior efficacy in the removal of gutta-percha and bioceramic sealer compared to 10% citric acid and 2% acetic acid. The apical third remained the most difficult region for complete removal of obturating material.

Introduction

Successful endodontic therapy relies on proper cleaning, shaping, and three-dimensional obturation of the root canal system.[1] The primary objective of obturation is to achieve a fluid-tight seal that prevents microbial leakage and reinfection. Despite advancements in

endodontic techniques and materials, failure of root canal treatment may still occur due to persistent infection, inadequate obturation, procedural errors, or coronal leakage.[2] In such situations, nonsurgical endodontic retreatment becomes necessary.

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The primary goal of retreatment is the complete removal of previous obturating materials so that the root canal system can be effectively disinfected and re-obturated. However, complete removal of obturating materials remains a challenge, particularly when bioceramic sealers are used.

Bioceramic sealers have gained considerable popularity in modern endodontics because of their superior biological and physical properties, including excellent biocompatibility, bioactivity, dimensional stability, and sealing ability.[3] These sealers penetrate dentinal tubules and form a chemical bond with dentin through hydroxyapatite formation, thereby creating a strong seal. Although advantageous during primary treatment, these same properties make their removal difficult during retreatment procedures.

Residual obturating material remaining on the canal walls may harbor microorganisms and compromise the success of retreatment.[4] Mechanical instrumentation alone is often insufficient to completely remove gutta-percha and bioceramic sealers, especially in the apical third and anatomical irregularities of the canal system. Therefore, chemical solvents are frequently used as adjuncts to facilitate removal.

Traditionally, solvents such as chloroform and xylene have been used for gutta-percha dissolution. However, concerns regarding toxicity, carcinogenic potential, and limited action on bioceramic sealers have encouraged the search for safer and more effective alternatives.

Weak organic acids have emerged as potential solvents because of their ability to chelate calcium ions and dissolve the inorganic components of bioceramic sealers.[5] Citric acid is a well-known chelating agent capable of removing smear layer and dissolving calcium-based materials. Maleic acid, a stronger organic acid with a lower pH, has demonstrated superior smear layer

removal and enhanced demineralization, especially in the apical third.[6] Acetic acid, though comparatively weaker, has also shown the ability to dissolve calcium-based materials through acid dissociation and chelation.

Considering the increasing use of bioceramic sealers and the challenges associated with their retreatment, this study was undertaken to comparatively evaluate the efficacy of 2% acetic acid, 10% citric acid, and 7% maleic acid in the removal of gutta-percha obturated with bioceramic sealer.

MATERIALS AND METHODS

Forty-five extracted human single-rooted teeth with fully formed apices were selected for the study. Teeth with fractures, resorption, calcification, or previously treated canals were excluded. The samples were cleaned and stored in 0.1% thymol solution until use.

Materials Used

- Extracted human single-rooted teeth (n = 45)

1. Endodontic Instruments

- K-files (#10 and #15)
- ProTaper Universal rotary files up to F3
- ProTaper retreatment files (D1, D2, D3)
- Endodontic hand instrument

2. Irrigants and Solutions

- 3% Sodium hypochlorite (NaOCl)
- 17% Ethylenediaminetetraacetic acid (EDTA)
- Normal saline

3. Obturating Materials

- Gutta-percha cones
- Bioceramic sealer

4. Solvents Used

- 2% Acetic acid
- 10% Citric acid
- 7% Maleic acid

Armamentarium

- Endodontic motor
- Irrigation syringes with side-vented needles
- Paper points
- Diamond disc for decoronation
- Chisel and mallet for splitting teeth

Methodology

Decoronation and Canal Preparation



Figure 1: Decoronation of extracted single-rooted teeth using a diamond disc to standardize root length.



Figure 2. Standardized root specimens obtained after decoronation

The crowns of all teeth were removed using a diamond disc to standardize root length as shown in Figure 1 and 2. Canal patency was established using #10 K-files, and working length was determined.

Biomechanical preparation was performed using ProTaper Universal rotary files up to F3 as shown in Figure 3. Irrigation was carried out using 3% sodium hypochlorite between each instrument. Final irrigation was done with 17% EDTA followed by normal saline.

Obturation

After drying the canals with paper points, all samples were obturated using gutta-percha cones and bioceramic sealer.

The specimens were then stored to allow complete setting of the sealer for 10 days. The samples were randomly divided into three groups according to the



Figure 3: Biomechanical preparation of root canals using ProTaper Universal rotary files up to F3

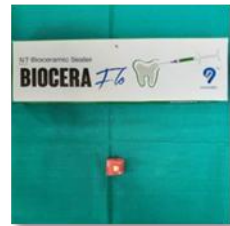


Figure 4: Obturation of prepared root canals using gutta-percha cones and bioceramic sealer

solvent used during retreatment:

- Group I: 2% Acetic acid
- Group II: 10% Citric acid
- Group III: 7% Maleic acid

Retreatment Procedure

- Retreatment was performed using ProTaper retreatment files (D1, D2, and D3) in conjunction with the respective solvents. (Figure 5)
- Solvents were introduced into the canals to soften the obturating material and facilitate removal. (Figure 6)
- A total of 2-3 ml of the solvent was used per sample during the retreatment and was kept for 3 minutes for coronal middle and apical third respectively.
- Following retreatment, the roots were longitudinally sectioned using a chisel and mallet.

Evaluation of Residual Material:

- The sectioned specimens (Figure 7) were examined under a stereomicroscope. And the amount of residual obturating material was assessed in the coronal, middle, and apical thirds.
- The percentage of remaining sealer was calculated using the formula:
- Percentage of remaining sealer = $(\text{Area of residual sealer} / \text{Total canal area}) \times 100$



Figure 5: Representative image of ProTaper Universal Retreatment File System (Dentsply Maillefer, Switzerland) used for removal of gutta-percha and bioceramic sealer during retreatment procedures.



Figure 6: Solvents used during retreatment: (A) 2% Acetic Acid, (B) 10% Citric Acid, and (C) 7% Maleic Acid.

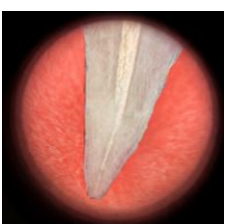


Figure 7: Decoronated and longitudinally sectioned specimens used for stereomicroscopic evaluation)

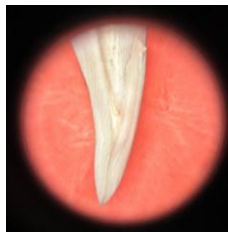
Stereomicroscope Images



8 (A)



8 (B)



8 (C)

Figure 8: Representative stereomicroscopic images showing residual obturating material after retreatment in the 2% Acetic Acid 8(A), 10% Citric Acid 8(B), and 7% Maleic Acid 8(C) groups.

RESULTS AND OBSERVATIONS

Table 1 demonstrates the intergroup comparison of the mean percentage of residual obturating material at the coronal, middle, apical, and overall canal levels among 2% acetic acid, 10% citric acid, and 7% maleic acid groups. A statistically significant difference was observed among the three groups at all canal regions ($p < 0.001$).

At the coronal third, the highest residual material was observed in the 2% acetic acid group (18.60 ± 1.25), followed by the 10% citric acid group (12.82 ± 0.78), whereas the lowest residual material was noted in the 7% maleic acid group (8.39 ± 0.51) ($F = 476.95$, $p < 0.001$). Similar findings were observed at the middle third, where the mean residual material was 22.54 ± 1.31 in the acetic acid group, 15.67 ± 0.71 in the citric acid group, and 9.90 ± 0.47 in the maleic acid group ($F = 730.24$, $p < 0.001$).

At the apical third, the highest amount of residual material was observed in all groups, with mean values of 27.59 ± 1.30 for 2% acetic acid, 20.36 ± 0.72 for 10% citric acid, and 13.68 ± 0.48 for 7% maleic acid ($F = 883.85$, $p < 0.001$). Similarly, the overall residual material was significantly highest in the acetic acid group (22.90 ± 1.26) and lowest in the maleic acid group (10.64 ± 0.48) ($F = 709.68$, $p < 0.001$).

Overall, Table 1 indicates that 7% maleic acid exhibited the least residual obturating material and therefore demonstrated superior efficacy in the removal of gutta-percha and bio ceramic sealer compared with 10% citric acid and 2% acetic acid. (Figure 9).

Table 2 presents the multiple intergroup comparisons using Tukey HSD post hoc analysis. The analysis revealed statistically significant pairwise differences between all the study groups at the coronal, middle, apical, and overall levels ($p < 0.001$).

At the coronal third, the mean difference between 2% acetic acid and 10% citric acid was 5.77, while the difference between 2% acetic acid and 7% maleic acid was 10.20. Similarly, the mean difference between 10% citric acid and 7% maleic acid was 4.43, indicating significantly lower residual material with maleic acid.

At the middle third, the highest mean difference was observed between 2% acetic acid and 7% maleic acid (12.64), followed by 2% acetic acid versus 10% citric acid

Table 1. Intergroup Comparison of Mean Percentage of Residual Material at Coronal, Middle, Apical, and Overall Levels among 2% Acetic Acid, 10% Citric Acid, and 7% Maleic Acid(n=45)

Region	Group	N	Mean \pm SD	SE	95% CI	Min–Max	F value	p value
Coronal Third	2% Acetic Acid	15	18.60 \pm 1.25	0.32	17.90–19.29	16.8–21.0	476.9	0.001*
	10% Citric Acid	15	12.82 \pm 0.78	0.20	12.38–13.26	11.7–14.1		
	7% Maleic Acid	15	8.39 \pm 0.51	0.13	8.11–8.67	7.6–9.2		
Middle Third	2% Acetic Acid	15	22.54 \pm 1.31	0.33	21.81–23.26	20.8–25.1	730.2	0.001*
	10% Citric Acid	15	15.67 \pm 0.71	0.18	15.27–16.06	14.7–16.9		
	7% Maleic Acid	15	9.90 \pm 0.47	0.12	9.63–10.16	9.2–10.8		
Apical Third	2% Acetic Acid	15	27.59 \pm 1.30	0.33	26.87–28.31	25.6–30.2	883.8	0.001*
	10% Citric Acid	15	20.36 \pm 0.72	0.18	19.96–20.76	19.3–21.7		
	7% Maleic Acid	15	13.68 \pm 0.48	0.12	13.41–13.95	12.9–14.5		
Overall Residual	2% Acetic Acid	15	22.90 \pm 1.26	0.32	22.20–23.60	21.1–25.4	709.6	0.001*
	10% Citric Acid	15	16.28 \pm 0.74	0.19	15.86–16.69	15.2–17.6		
	7% Maleic Acid	15	10.64 \pm 0.48	0.12	10.38–10.91	9.9–11.5		

* Statistically significant at $p < 0.05$ using one-way ANOVA, SE: Standard Error; CI: Confidence Interval.

Figure 9. Comparison of Mean Percentage of Residual Obturating Material among 2% Acetic Acid, 10% Citric Acid, and 7% Maleic Acid at Coronal, Middle, and Apical Thirds of the Root Canal System

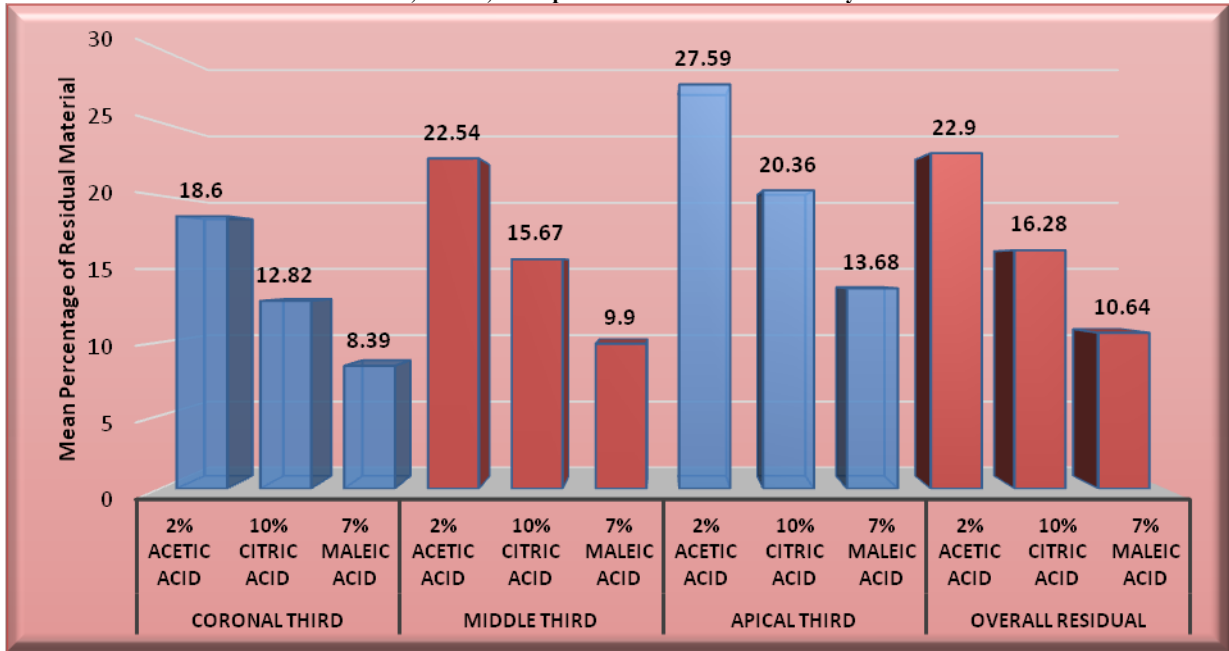


Table 2. Multiple Intergroup Comparison of Mean Percentage of Residual Material at Coronal, Middle, Apical, and Overall Levels using Tukey HSD Post Hoc Test

Region	Group Comparison	Mean Difference	SE	p value	95% CI
Coronal Third	2% Acetic Acid vs 10% Citric Acid	5.77	0.33	0.001*	4.96–6.57
	2% Acetic Acid vs 7% Maleic Acid	10.20	0.33	0.001*	9.40–11.01
	10% Citric Acid vs 7% Maleic Acid	4.43	0.33	0.001*	3.62–5.23
Middle Third	2% Acetic Acid vs 10% Citric Acid	6.86	0.33	0.001*	6.06–7.67
	2% Acetic Acid vs 7% Maleic Acid	12.64	0.33	0.001*	11.83–13.44
	10% Citric Acid vs 7% Maleic Acid	5.77	0.33	0.001*	4.96–6.57
Apical Third	2% Acetic Acid vs 10% Citric Acid	7.22	0.33	0.001*	6.42–8.03
	2% Acetic Acid vs 7% Maleic Acid	13.90	0.33	0.001*	13.10–14.71

	10% Citric Acid vs 7% Maleic Acid	6.68	0.33	0.001*	5.87–7.48
Overall Residual	2% Acetic Acid vs 10% Citric Acid	6.62	0.32	0.001*	5.83–7.41
	2% Acetic Acid vs 7% Maleic Acid	12.26	0.32	0.001*	11.46–13.05
	10% Citric Acid vs 7% Maleic Acid	5.63	0.32	0.001*	4.84–6.42

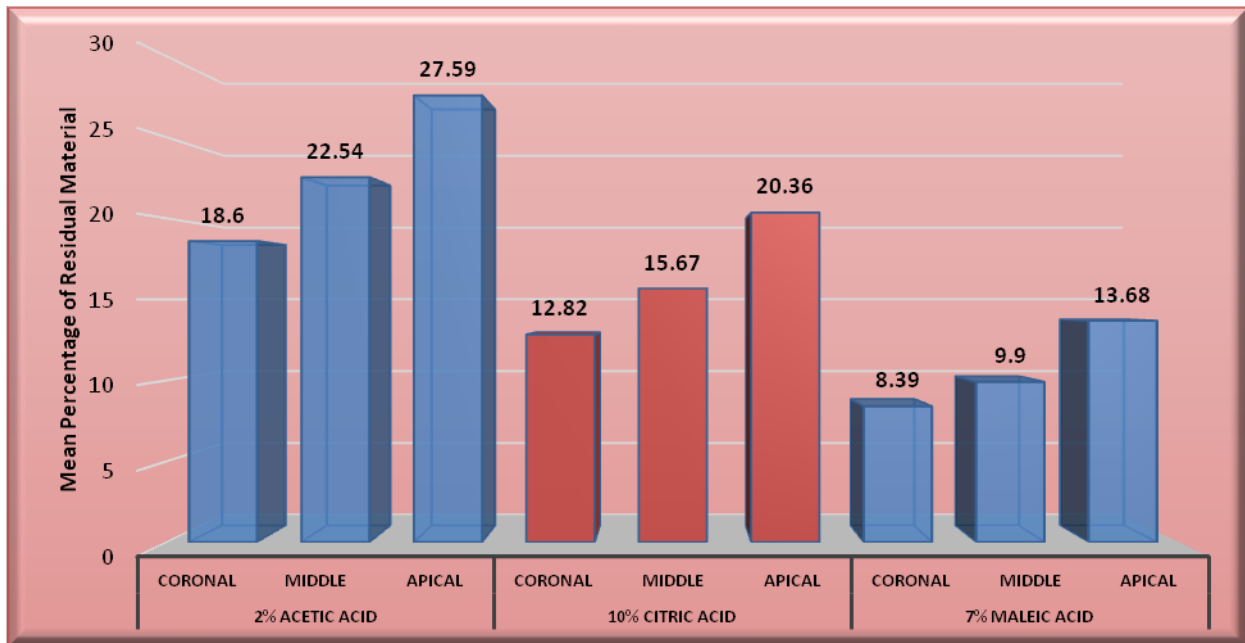
* Statistically significant at $p < 0.05$ using one-way ANOVA, SE: Standard Error; CI: Confidence Interval.

Table 3. Intragroup Comparison of Mean Percentage of Residual Material at Coronal, Middle, and Apical Thirds within the Study Groups (n=45)

Group	Region	n	Mean \pm SD	SE	95% CI	Min–Max	F value	p value	Partial Eta Squared
2% Acetic Acid	Coronal	15	18.60 \pm 1.25	0.32	17.90–19.29	16.8–21.0	3753.27	0.001*	0.996
	Middle	15	22.54 \pm 1.31	0.33	21.81–23.26	20.8–25.1			
	Apical	15	27.59 \pm 1.30	0.33	26.87–28.31	25.6–30.2			
10% Citric Acid	Coronal	15	12.82 \pm 0.78	0.20	12.38–13.26	11.7–14.1	11119.9	0.001*	0.999
	Middle	15	15.67 \pm 0.71	0.18	15.27–16.06	14.7–16.9			
	Apical	15	20.36 \pm 0.72	0.18	19.96–20.76	19.3–21.7			
7% Maleic Acid	Coronal	15	8.39 \pm 0.51	0.13	8.11–8.67	7.6–9.2	29658.0	0.001*	1.000
	Middle	15	9.90 \pm 0.47	0.12	9.63–10.16	9.2–10.8			
	Apical	15	13.68 \pm 0.48	0.12	13.41–13.95	12.9–14.5			

Repeated Measures ANOVA; statistically significant at $p < 0.05$

Figure 10. Intragroup Comparison of Mean Percentage of Residual Obturating Material at Coronal, Middle, and Apical Thirds within the 2% Acetic Acid, 10% Citric Acid, and 7% Maleic Acid Groups (n=45)



(6.86). Likewise, at the apical third, the mean difference between acetic acid and maleic acid was 13.90, demonstrating markedly superior cleaning efficacy of 7% maleic acid in the apical region.

For overall residual material, significant differences were observed between all groups, with the greatest difference noted between 2% acetic acid and 7% maleic acid (12.26). These findings confirm that all three solvents differed significantly in their cleaning efficacy, with 7% maleic acid showing the best performance and 2% acetic acid showing the least effectiveness.

Table 3 depicts the intragroup comparison of residual obturating material among the coronal, middle, and apical thirds within each solvent group using repeated measures ANOVA. A statistically significant difference was observed among the canal regions within all the study groups ($p < 0.001$).

In the 2% acetic acid group, the mean residual material increased progressively from the coronal third (18.60 ± 1.25) to the middle third (22.54 ± 1.31) and reached the highest value in the apical third (27.59 ± 1.30). Repeated

measures ANOVA revealed a highly significant difference among the three canal regions ($F = 3753.27$, $p < 0.001$).

Similarly, in the 10% citric acid group, the mean residual material increased from 12.82 ± 0.78 at the coronal third to 15.67 ± 0.71 at the middle third and 20.36 ± 0.72 at the apical third ($F = 11119.92$, $p < 0.001$).

The 7% maleic acid group demonstrated the lowest residual material values at all canal levels, with mean values of 8.39 ± 0.51 , 9.90 ± 0.47 , and 13.68 ± 0.48 at the coronal, middle, and apical thirds, respectively. The intragroup comparison also showed highly significant differences among canal regions ($F = 29658.02$, $p < 0.001$).

The partial eta squared values indicated a very large effect size, suggesting that the canal region had a strong influence on the amount of residual obturating material remaining after retreatment. Overall, Table 3 indicates that the apical third consistently exhibited the highest residual obturating material irrespective of the solvent used, suggesting greater difficulty in achieving complete cleaning in the apical portion of the root canal system. Among the tested solvents, 7% maleic acid showed

superior efficacy with the least residual material at all canal levels. (Figure 10)

Statistical Analysis

The collected data were analyzed using IBM SPSS Statistics software version 30.0 (IBM Corp., Armonk, NY, USA). Normality of data distribution was assessed using the Shapiro–Wilk test, which confirmed normal distribution of the data ($p > 0.05$). Intergroup comparisons among the three solvent groups were performed using one-way analysis of variance (ANOVA) followed by Tukey’s post hoc test for multiple pairwise comparisons. Intragroup comparisons among the coronal, middle, and apical thirds within each group were analyzed using repeated measures ANOVA. A p -value of <0.05 was considered statistically significant.

Discussion

The success of nonsurgical endodontic retreatment depends largely on the effective removal of existing obturating materials from the root canal system. Residual filling material may harbor microorganisms and interfere with proper disinfection and re-obturation.[4]

Bioceramic sealers are increasingly used because of their excellent sealing ability, dimensional stability, and bioactivity.[3] However, these same properties contribute to the difficulty encountered during retreatment procedures.

In the present study, three weak organic acids—2% acetic acid, 10% citric acid, and 7% maleic acid—were evaluated for their efficacy in removing gutta-percha obturated with bioceramic sealer.

Among the tested solvents, 7% maleic acid demonstrated the highest efficacy. This superior performance may be attributed to its strong acidic nature, lower pH, and greater demineralizing potential.[6] Maleic acid effectively dissolves the inorganic components of the bioceramic sealer and enhances smear layer removal, particularly in the apical region.

Citric acid demonstrated moderate efficacy in removing obturating material. Its chelating action enables the dissolution of calcium ions present in bioceramic sealers, thereby weakening the sealer matrix and facilitating removal. However, its effectiveness was comparatively lower than maleic acid, possibly because of differences in pH, concentration, and penetration ability.

Acetic acid showed the least efficacy among the three groups. Although it possesses chelating properties and can dissolve calcium-based materials, its weaker acidic strength and lower concentration may have limited its effectiveness against the bioceramic sealer matrix.

An important finding of this study was that the maximum residual material was consistently observed in the apical third across all groups. This can be attributed to the complex anatomy, narrow diameter, and limited accessibility of the apical region, which hinder both mechanical instrumentation and solvent penetration.[7]

The findings of this study are consistent with previous literature, which reports that the apical third remains the most challenging area for complete removal of obturating materials during retreatment.

The efficacy of solvents is influenced not only by their chemical composition but also by physical properties such as surface tension, viscosity, and penetration ability. Solvents with lower surface tension and better flow characteristics are more capable of penetrating into dentinal tubules and irregularities, thereby enhancing dissolution of obturating material.

The use of weak organic acids in retreatment procedures may also offer advantages in terms of reduced cytotoxicity and improved biocompatibility compared to traditional solvents such as chloroform and xylene.

Despite the significant findings, the present study has certain limitations. Being an in-vitro study, it does not completely simulate clinical conditions such as variations in canal anatomy, presence of vital tissues, and intraoral environmental factors. In addition, evaluation under a stereomicroscope may not provide the same degree of detail as advanced imaging techniques such as micro-CT.[8]

Further clinical and laboratory studies are recommended to evaluate the long-term effectiveness and safety of these solvents during retreatment procedures.

CONCLUSION

- Within the limitations of this in-vitro study, the following conclusions can be drawn:
- None of the tested solvents achieved complete removal of gutta-percha and bioceramic sealer.
- 7% maleic acid demonstrated the highest efficacy in removing obturating material.
- 10% citric acid showed moderate efficacy.
- 2% acetic acid exhibited the least efficacy among the tested solvents.
- Statistically significant differences were observed

among all three groups ($p < 0.05$).

- The apical third consistently showed the highest amount of residual material.
- Appropriate solvent selection can improve the efficiency of endodontic retreatment procedures.

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