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Research Report

Efficacy of white vinegar, 37% phosphoric acid and 10% hydrofluoric acid on the retrievability of bioceramic sealer: an in-vitro study

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ABSTRACT

Background: *Root canal retreatment is performed when root canal treatment fails. One of the challenges in retreatment is the removal of root canal filling material. Bioceramic sealer is a new endodontic biomaterial that has many advantages. Nevertheless, the difficulty in retrieving bioceramic sealer in failed root canal treatment remains a controversy.* ***Purpose:*** *To investigate the efficacy of white vinegar, 37% phosphoric acid and 10% hydrofluoric acid in retrieving bioceramic sealer.* ***Methods:*** *Bioceramic sealer specimen was used as samples in this study consisting of 60 samples divided into four treatment groups. All groups were soaked in white vinegar, 37% phosphoric acid, 10% hydrofluoric acid and saline as negative control for 10 minutes. Microhardness testing was performed using Vickers Microhardness Tester, then all data were statistically analysed using One Way ANOVA and post-hoc LSD.* ***Results:*** *The result of this study showed mean and standard deviation of VHN of bioceramic sealer specimen which were soaked in white vinegar, 37% phosphoric acid, 10% hydrofluoric acid and saline as follows respectively: 45.11±2.39; 51.46±3.64; 29.77±2.66; 66.16±1.70 with p value <0.001.* ***Conclusion:*** *10% hydrofluoric acid has the greatest potency as root canal solvent for bioceramic sealer during root canal retreatment.*

Keywords: *Acid, bioceramic sealer, retreatment, retrievability, VHN*

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INTRODUCTION

Root canal treatment is indicated in cases of pulp disorders, namely irreversible pulpitis and pulp necrosis, with or without the presence of periapical abnormalities.[1] The main goal of root canal treatment is to clean the root canal of pathogenic microorganisms and prevent reinfection by performing adequate chemo-mechanical preparation and hermetic obturation of the root canal.[2] Root canal treatment is a procedure aimed at preserving natural teeth, with a fairly high success rate of up to 97%.[3] Root canal treatment is performed according to the principles of the endodontic triad and is followed by proper coronal sealing.[4] The endodontic triad consists of three stages: access preparation, cleaning and shaping, and obturation.[5]

Good obturation is the key to the success of root canal treatment.[6] The main purpose of obturation is to fill the prepared root pulp space to achieve a three-dimensional hermetic seal and prevent bacterial activity from accessing the periapical tissue.[7] The determining factors for the success of obturation itself depend on the technique and materials used in the obturation.[7] The obturation technique that combines solid materials such as gutta-percha and semi-solid materials like sealer has become the standard protocol in endodontic treatment.[8] Several types of sealers that are commonly used are classified into five groups based on their composition, namely: calcium hydroxide-based, zinc oxide eugenol (ZOE) based, resin-based, glass ionomer-based, and bioceramic sealers.[9]

Bioceramic sealers are relatively new biomaterials in the field of endodontics, with increasing popularity year by year due to their many advantages over traditional sealers in general.[10] This sealer is a combination of ceramics that are biocompatible and obtained in situ and in vivo through various stages of chemical processes.[11] Based on its chemical composition, bioceramic sealers are classified into mineral trioxide-based sealers, calcium phosphate-based sealers, calcium silicate-based sealers, and phosphate-based sealers.[12] The advantages of bioceramic sealers include being biocompatible, bioactive, not easily soluble, having a good flow rate, not prone to shrinkage, antibacterial, and capable of promoting the healing of periapical lesions.[13] Although bioceramic sealers have many advantages, the issue of solubility of these sealers during retreatment of root canals remains a controversy.[14]

Retreatment of root canals is performed after a failure occurs post root canal treatment.[15] Although the success rate of root canal treatment is quite high, failure can occur due to several factors such as secondary or persistent infections, coronal leakage, marginal periodontal disease, missing canals, cracks, root fractures, and root perforations.[16] The success of retreatment of root canals depends on the ability to remove the filling material from previous treatments in order to gain maximum access to the apical third of the root canal for debridement and re-obturation.[17] The success rate of root canal retreatment can significantly decrease from 87% to 47% if the filling material cannot be adequately cleaned, leaving residues that obstruct the contact of irrigating solutions and intracanal medications with the dentin tubules.[18] The aim of this study is to evaluate and compare the efficacy of white vinegar, phosphoric acid 37% and hydrofluoric acid 10% on the retrieval of bioceramic sealer.

MATERIALS AND METHODS

This was an in-vitro laboratory experimental research with post-test control group design. The sample in this study is a bioceramic sealer branded C-Root SP (China), which was cast in a silicone mould shaped like a hexagonal prism with a side length of 3 mm and a height of 1 mm (Figure 1A). The mould is then placed in an incubator set at a temperature of 37°C and 100% humidity for 3x24 hours (Figure 1B). The treatment groups being studied are white vinegar, phosphoric acid 37%, hydrofluoric acid 10% and saline. The total number of samples is set at 15 samples per group. Thus, the total sample for the four treatment groups is 60 samples.

The tools used in this study incudes a silicone mould in the shape of a hexagonal prism with a side length of 3 mm and a height of 1 mm, Vickers Microhardness Tester (Future-Tech Corp, Japan), incubator (Sanyo, Japan), plastic container (Tupperware, USA), adhesive label (3M, USA), mask (Evo, Germany), tweezers (Osung, Korea), micropipette (Transferpette® S, Germany), Chip blower (Schezher, Germany). The materials used in this research are bioceramic sealer (C-Root SP, China), white vinegar (Dixi, Indonesia), phosphoric acid 37% (Any-Etch HV, Korea), hydrofluoric acid 10% (DSP Porcelain, Brazil) and saline (MJB Pharma, Indonesia).

Figure 1. Bioceramic sealer casted in a silicone mould (A). The mould placed in an incubator (B).

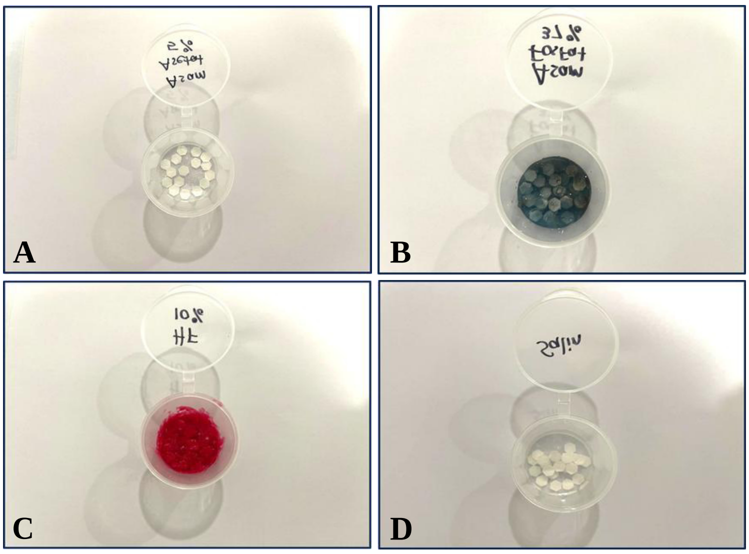

Figure 2. Specimens soaked in 10 ml white vinegar (A). Specimens soaked in 10 ml phosphoric acid 37% (B). Specimens soaked in 10 ml hydrofluoric acid 10% (C). Specimens soaked in 10 ml saline (D).

The first plastic container contains 10 ml of white vinegar and is labelled as group I. The second plastic container contains 10 ml of 37% phosphoric acid and is labeled as group II, the third plastic container contains 10 ml of 10% hydrofluoric acid and is labeled as group III, and the fourth plastic container contains 10 ml of saline and is labeled as group IV. After the bioceramic sealer has been allowed to set for 3x24 hours, the bioceramic sealer specimens are removed from the silicone moulds. A total of 15 bioceramic sealer specimens are transferred with tweezers and soaked in a plastic container of group I (Figure 2A). Next, 15 bioceramic sealer specimens are transferred with tweezers and soaked in a plastic container of group II (Figure 2B). Then, 15 bioceramic sealer specimens are transferred with tweezers and soaked in a plastic container of group III (Figure 2C). Subsequently, 15 bioceramic sealer specimens are transferred with tweezers and soaked in a plastic container of group IV (Figure 2D). After 10 minutes of soaking, all specimens are removed with tweezers and dried by placing them on a dry tissue paper and then sprayed with a chip blower. Each specimen is transferred into a new and dry plastic container, sealed tightly, and labeled with a sample number.

The specimens were studied with a light microscope under 40x magnification. Defective or crack samples were excluded from this study. After samples polishing, surface microhardness test was performed using a Vickers Microhardness Tester (Future-Tech Corp, Japan) as seen in Figure 7. A 50-g load and a dwell time diamond indenter were used for 10 seconds. Three indentations were created on the polished surface of each sample at different locations according to ASTM E384 standard for Vickers microhardness test. The Vickers microhardness number (VHN) was calculated with the following formula: VHN=1/4 1:854xL/d^2 where L is the applied load (kg) and d is the mean indentation diagonal length (mm). The statistical techniques used in this study include One-Way Variance Analysis (ANOVA) and post-hoc LSD analysis for data analysis.

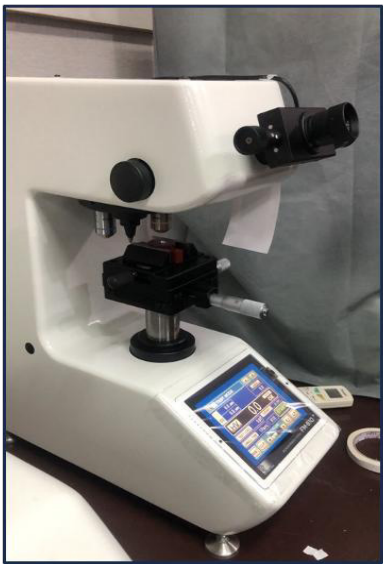

Figure 3. Microhardness test performed using a Vickers Microhardness Device (Future-Tech Corp, Japan).

Table 1. The average of microhardness value (VHN) of the bioceramic sealer subjected to four treatment groups

Group	Vickers Hardness Number Means±SD
White vinegar	45.11±2.39
Phosphoric acid 37%	51.46±3.64
Hydrofluoric acid 10%	29.77±2.66
Saline	66.16±1.70

Table 2. Results of normality and homogeneity tests

Group	Normality *p*	Homogenity *p*
White vinegar	0.292	0.082
Phosphoric acid 37%	0.884	
Hydrofluoric acid 10%	0.672	
Saline	0.103	

Table 3. The results of the statistical test on the effectiveness of white vinegar, 37% phosphoric acid, 10% hydrofluoric acid, and saline on the retrievability of bioceramic sealer

Group	Mean±SD	*p*
White vinegar	45.11±2.39	<0.001
Phosphoric acid 37%	51.46±3.64	
Hydrofluoric acid 10%	29.77±2.66	
Salin	66.16±1.70	

RESULTS

Table 1 describes the mean and standard deviation of microhardness (VHN) values of the bioceramic sealer treated with white vinegar, 37% phosphoric acid, 10% fluoride acid, and saline, which are 45.11±2.39; 51.46±3.64; 29.77±2.66; and 66.16±1.70, respectively.

The graph in Figure 4 shows that saline treatment of the bioceramic sealer resulted in the highest average microhardness value (VHN) of 66.16±1.70. The treatment with 37% phosphoric acid on the bioceramic sealer showed the second highest average microhardness value (VHN) of 51.46±3.64. This was followed by the treatment with white vinegar on the bioceramic sealer, which showed an average microhardness value (VHN) of 45.11±2.39. The treatment with 10% fluoride acid on the bioceramic sealer had the lowest average microhardness value (VHN) of 29.77±2.66.

Table 2 describes the normality test using Shapiro-Wilk and the homogeneity test using Levene's Test. The results of the Shapiro-Wilk normality test for the groups of white vinegar, 37% phosphoric acid, 10% hydrofluoric acid, and saline showed a p-value > 0.05, which means that the data is distributed normally. Meanwhile, the results of the Levene's homogeneity test also showed a p-value > 0.05, indicating that the data is homogeneous. Since the research data is normally distributed and homogeneous, data analysis can proceed using one-way ANOVA and post hoc LSD statistical tests.

Table 3 describes the results of the one-way ANOVA statistical test showed a p-value of <0.001 ($p \leq 0.05$),

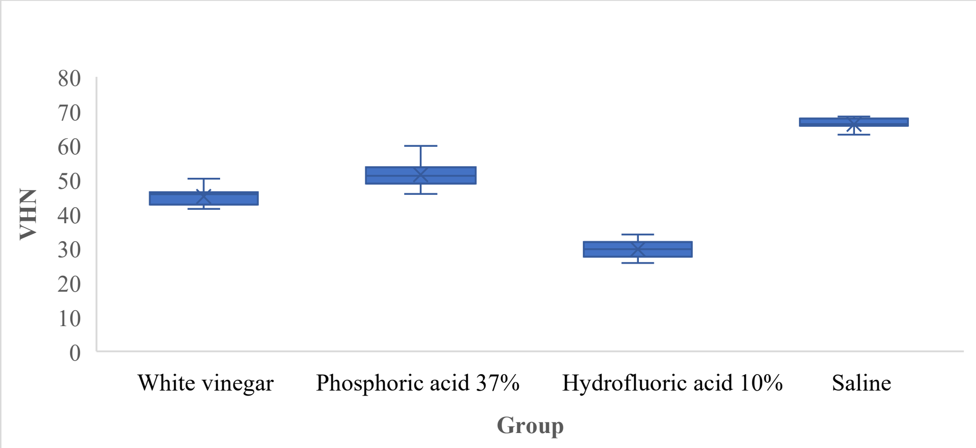

Figure 4. The graph average of microhardness value (VHN) of the bioceramic sealer subjected to four treatment groups.

Table 4. Results of the post hoc LSD test

Group		Mean Difference	p
White vinegar	Phosphoric acid 37%	-6.34733*	<.001
	Hydrofluoric acid 10%	15.34000*	<.001
	Saline	-21.05667*	<.001
Phosphoric acid 37%	White vinegar	6.34733*	<.001
	Hydrofluoric acid 10%	21.68733*	<.001
	Saline	-14.70933*	<.001
Hydrofluoric acid 10%	White vinegar	-15.34000*	<.001
	Phosphoric acid 37%	-21.68733*	<.001
	Saline	-36.39667*	<.001
Saline	White vinegar	21.05667*	<.001
	Phosphoric acid 37%	14.70933*	<.001
	Hydrofluoric acid 10%	36.39667*	<.001

indicating that there is a significant difference in the mean microhardness values (VHN) of the bioceramic sealer treated with white vinegar, 37% phosphoric acid, 10% fluoride acid, and saline. From this research, it can be stated that there are differences in the efficacy of white vinegar, 37% phosphoric acid, and 10% fluoride acid on the solubility of the bioceramic sealer ($p \leq 0.05$).

Table 4 describes the results of the post hoc LSD test indicate that there are significant differences in efficacy among the four treatment groups as follows: First, between white vinegar and 37% phosphoric acid, 10% hydrofluoric acid, and saline. Second, between 37% phosphoric acid and white vinegar, 10% hydrofluoric acid, and saline. Third, between 10% hydrofluoric acid and white vinegar, 37% phosphoric acid, and saline. Fourth, between saline and white vinegar, 37% phosphoric acid, and 10% hydrofluoric acid.

DISCUSSION

Acid solutions have been reported to reduce the remaining bioceramic sealer, particularly in the isthmus area and dentin tubules that are difficult to clean with mechanical instruments.[18] Several in-vitro studies have shown the efficacy of hydrochloric acid[19] acetic acid[18] glycolic acid[20] maleic acid[21] citric acid[22] and phosphoric acid[23] on the solubility of bioceramic sealers. Several studies also show that solutions with low pH can affect the tensile strength, surface roughness, and push-out bond strength of bioceramic sealers because these solutions can cause porosity and voids.[24] An acidic environment can lead to corrosion due to the decomposition of calcium sulfoaluminate and calcium hydroxide phases.[25]

The results of the one-way ANOVA study indicate that white vinegar, 37% phosphoric acid, and 10% hydrofluoric acid are effective in retrieving bioceramic sealers, as evidenced by a significant difference ($p<0.001$) compared to the saline group as a negative control. The results of this study indicate that the treatment of 10% hydrofluoric acid on the bioceramic sealer shows the greatest solubility effect, with the lowest average microhardness value (VHN) among the treatment groups, which is 29.77±2.66. The smaller the microhardness value (VHN), the greater the solubility effect of the test material on the bioceramic sealer.

Based on the research conducted by Samimi et al. (2018), 9% hydrofluoric acid applied for 90 seconds produces a porous surface on Pro-Root MTA, which contains calcium silicate. Other research has also reported that hydrofluoric acid is effective in the demineralization process of dental ceramics containing silicate.[26,27] Silicate is the main component of bioceramic sealer.[28] However, until now, there has been no research on the effects of fluoride acid on the solubility of bioceramic sealers, so this study has novelty value. The use of hydrofluoric acid in root canals

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must be performed with maximum isolation using a rubber dam. This is because hydrofluoric acid has toxicity when in contact with the oral mucosa that can cause tissue damage.[29]

The results of this study also show that white vinegar containing 5% acetic acid can retrieve bioceramic sealer, where the average microhardness value (VHN) of the bioceramic sealer treated with white vinegar is 45.11±2.39. This result is in line with previous studies that demonstrate the efficacy of acetic acid in retrieving bioceramic sealers.[18,30,31]

Carillo's research reported that the solubility of bioceramic sealer in white vinegar is better compared to 6% NaOCl and carbonated water, measured by the achievement of patency during retreatment of root canals.[30] Inderpal's study found that white vinegar is more effective in retrieving bioceramic sealer compared to 37% phosphoric acid and chloroform, measured by the working length achieved during root canal retreatment.[18] The research by Abraham et al., reported that 2% acetic acid is more effective in retrieving bioceramic sealer compared to 2% carbonic acid, as measured by microhardness testing.[31]

This study indicates phosphoric acid is effective in retrieving bioceramic sealer, where the average microhardness value (VHN) of the bioceramic sealer treated with 37% phosphoric acid is 45.11±2.39. These results are consistent with past studies that demonstrate the efficacy of phosphoric acid in retrieving bioceramic sealers.[18,23,32] The study by Lee et al., reported that exposure to 37% phosphoric acid corroded the crystalline structure on the surface of the bioceramic sealer and created a cracked surface with internal porosity.[23] The research by Samimi et al., noted the effects of 37% phosphoric acid on changes in the surface characteristics of bioceramic materials, showing an irregular structure and the loss of spindle-shaped features in scanning electron microscope (SEM) tests.[32] The study by Kayahan et al. reported the effects of 37% phosphoric acid on the reduction of compressive strength of the bioceramic sealer using a universal testing machine.[33] 37% phosphoric acid can cause ulceration of periodontal tissue and damage to the oral mucosa, so the use of 37% phosphoric acid in the root canal must be done with rubber dam isolation.[34]

The results of the post hoc LSD test indicate that there are significant differences among all treatment groups ($p<0.001$). The efficacy of 10% hydrofluoric acid in retrieving bioceramic sealer is better than that of white vinegar and 37% phosphoric acid, with a significant difference. This is due to the ability of hydrofluoric acid to retrieve the silicate particles found in dental.[26,27,28] Silicate is the main component found in bioceramic sealers[28], so hydrofluoric acid can also retrieve bioceramic sealers.

The efficacy of white vinegar containing 5% acetic acid in retrieving bioceramic sealer is better than that of 37% phosphoric acid. This research finding is supported by a study conducted by[31], which found that acetic acid is the most effective in reducing the microhardness of bioceramic materials. Another study conducted by Inderpal also showed that the use of 5% acetic acid is more effective than 37% phosphoric acid and chloroform in restoring the patency of root canals and the working length of all samples studied when performing retreatment of root canals filled with bioceramic sealer.[18]

The efficacy of 37% phosphoric acid is better than the saline group as a negative control in retrieving bioceramic sealer. These results are in line with the research by Lee et al., which reported that the application of 37% phosphoric acid can etch and create porosity in calcium-based sealers.[23] The study by Samimi et al., reported that the effect of 37% phosphoric acid on the surface characteristics of bioceramic materials can retrieve both crystalline and amorphous structures, resulting in a rough and porous surface.[32] Research by Kayahan et al., indicated that the application of 37% phosphoric acid on bioceramic materials can lead to porosity and a decrease in push-out bond strength, compressive strength, and microhardness.[33]

Another method that could be used to better observe the solubility of bioceramic sealers is by conducting clinical simulations on real human teeth, followed by examination using scanning electron microscopy. (SEM). In addition to the chemical method using solvent materials, a combination with mechanical methods can also be performed, specifically using mechanical instruments such as retreatment files and/or ultrasonic devices to assess the efficacy of the removal of bioceramic sealers to the fullest extent.

There has not yet been any research comparing the time variable of solvent materials in assessing the efficacy on the solubility of bioceramic sealers. However, soaking for more than 10 minutes may indicate a higher efficacy of bioceramic sealer solubility. This is based on research by Aiswarya et al., 2023, which shows that solvents such as xylene, thyme oil, and orange oil are more effective in retrieving Roekoseal, AH Plus sealer, and MTA Fillapex in 10 minutes compared to 5 minutes and 2 minutes.[35]

In conclusion, there is efficacy of white vinegar, 37% phosphoric acid, and 10% hydrofluoric acid on the solubility of bioceramic sealer. The 10% hydrofluoric acid is more effective in retrieving bioceramic sealer compared to white vinegar, while white vinegar is more effective in retrieving bioceramic sealer compared to 37% phosphoric acid.

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