

Effect of different final irrigation solutions on push-out bond strength of root canal filling material

Rahmatillah,¹ Isyana Erlita¹ and Buyung Maglenda²

¹Department of Conservative Dentistry, Faculty of Dentistry, Universitas Lambung Mangkurat

²Endodontist, Sultan Suriansyah General Hospital

Banjarmasin – Indonesia

ABSTRACT

Background: The adhesion of root canal filling material to dentin is one of the crucial factors in determining the success of endodontic treatment. However, the smear layer that forms during instrumentation serves as an interface that impedes the bonding mechanism of the filling material. A proper irrigation solution is required to remove the smear layer and provide a dentin surface that supports the bonding mechanism of the filling material in establishing good adhesion. **Purpose:** This study aims to evaluate and compare the bond strength of filling material with different final irrigation solutions. **Methods:** Mandibular premolars were prepared by a crown down, pressure-less technique and divided into three final irrigation groups (2.5% NaOCl, 17% EDTA and 20% citric acid). The root canal of each tooth was obturated using epoxy sealer and gutta-percha. A two-millimetre-thick section of the apical third portion of each group was arranged for the push-out assessment using a universal testing machine in an apical to coronal direction at 1 mm/min crosshead speed. **Results:** A one-way ANOVA test indicated the difference in push-out bond strength among the groups ($p < 0.05$). A post hoc Bonferroni test presented a statistically significant difference in the bond-strength value between the 2.5% NaOCl group compared with the 20% CA group ($p < 0.05$). **Conclusion:** The push-out bond strength of root canal filling material is increased by applying a chelating agent as the final irrigation solution where 20% of CA presents the highest push-out bond strength.

Keywords: 20% citric acid; 17% EDTA; 2.5% NaOCl; push-out bond strength

Correspondence: Rahmatillah, Department of Conservative Dentistry, Faculty of Dentistry, Universitas Lambung Mangkurat, Jl. Veteran 128B, Banjarmasin 70122, Indonesia. Email: rahmatillahaz@gmail.com

INTRODUCTION

The bonding ability to dentin is a crucial feature of the root canal filling material. The material, which is frequently used as a root canal filling, is gutta-percha. Nevertheless, gutta-percha must be combined with a root canal sealer since gutta-percha does not adhere to root canal dentin.¹ There are two main concerns regarding material adhesion to root canal dentin. In static situations, the adhesion would prevent fluid percolation between the filling material and the root canal dentin. In dynamic situations, it would prevent dislodgement of the filling material from the root canal during subsequent manipulations, thereby reducing the risk of contamination.²

The smear layer serves as an interface between the root canal filling material and the dentin.³ The removal of the

smear layer advances sealer adhesion and affects the bond strength of the AH Plus sealer.^{4,5} Smear layer removal increases the contact area and the sealing ability of the sealer, so it produces better adaptation. Smear layer removal enables the sealer tags extension to the dentin tubules, which results in the formation of mechanical locking and efficient micro-retention.^{5,6} The sealer contact to the dentin also becomes closer, so it optimises the adhesion due to the formation of chemical bonds.⁷

Ethylenediaminetetraacetic acid (EDTA) is suggested as an irrigation solution because of its nature as a chelating agent. This irrigation solution has the ability to eliminate the inorganic portions of the smear layer. However, EDTA that is used as a single irrigation solution is not effective to eliminate the smear layer entirely.⁸ A proteolytic agent, one of which can be sodium hypochlorite (NaOCl), must

be used to eliminate the organic portion of the smear layer. NaOCl and EDTA irrigation solutions can remove the inorganic portions of the smear layer and expose collagen fibres. Moreover, the collagen fibres serve as a substrate for sealer infiltration and hybrid layer formation.^{8,9} A combination of NaOCl with a chelating agent or acidic material is required to eliminate both organic and inorganic portions of the smear layer. Consequently, it has been recommended to apply NaOCl along with EDTA or citric acid for irrigation procedures.¹⁰

Citric acid has been recommended as an alternative chelating agent. The effectiveness of 10%–50% citric acid in removing inorganic portions of the smear layer has been evaluated.¹¹ Olivieri *et al.*¹¹ reported that citric acid has a more effective smear layer removal effect in the apical and middle third root canals compared to EDTA 17%. Besides, Prado *et al.*¹² showed that both EDTA and citric acid are more effective in eliminating the smear layer at the apical third with a three-minute application.

The push-out test has been described as one of the most reliable, accurate, effective, and easy methods to measure the bond value between the sealer, dentin, and core material. Likewise, the push-out test can evaluate the bond strength to a low value at various depths of root canal dentin.^{8,13} The current study was performed to analyse the push-out bond strength between gutta-percha and epoxy resin sealer to dentin with the final irrigation solution 17% EDTA and 20% citric acid.

MATERIALS AND METHODS

The ethics committee of the Dentistry Faculty, Universitas Lambung Mangkurat No. 023 / KEPKG-FKGULM/EC/I/2020 approved this research and declared it to be clear from any ethical issues. This study used a post-test only with a control group design. The samples were 21 premolar teeth with the following inclusion criteria: mandibular premolar teeth extracted due to orthodontic treatment, straight and perfectly formed apex, and no root fractures. Teeth with caries, root morphological anomalies, and more than one root canal were excluded.

Root canal treatment was carried out on the selected teeth. The teeth were cut through the cemento-enamel junction with a double-sided, diamond disk (Suzhou Sydent Tools Co., Ltd, Suzhou, China) to leave a 14 mm root section with a working length of 13 mm.^{13,14} Preparation of the root canal was performed by a crown down, pressure-less technique with ProTaper hand-use instruments (Dentsply Maillefer, Ballaigues, Switzerland). Preparation was initiated by K-file #10 (Dentsply Maillefer, Ballaigues, Switzerland) throughout 2/3 of the working length. Thereafter, the preparation was carried out with S1 and S2 files according to the working length for widening the 2/3 coronal portion. Furthermore, a 1/3 apical portion preparation was performed with F1, F2, and

F3 files according to the working length (according to the manufacturer's instructions).

All of the root canals were irrigated with a 3 ml 2.5% NaOCl solution during instrumentation for each file size up to the F3 file (size 30, 0.09 taper). At the end of instrumentation, the root canals were randomly divided into three final irrigation groups, namely Group I (5 ml 2.5% NaOCl), Group II (5 ml 17% EDTA), and Group III (5 ml 20% citric acid). The irrigation was carried out using a 30 G, close-end, single side, vent needle (OneMed, Sidoarjo, Indonesia) for three minutes.^{15,16} Irrigation was done by a manual, dynamic-agitation technique (hand-activated, well-fitting, #F2 gutta-percha (Dentsply Maillefer, Ballaigues, Switzerland)) with push-pull movement 100 times/30 seconds.¹⁷ Thereafter, a paper point was inserted to dry up the root canal.¹⁸

The obturation was performed by manipulating the sealer (AH Plus, Dentsply, DeTrey GmbH, Konstanz, Germany) and ProTaper #F3 gutta-percha (Dentsply Maillefer, Ballaigues, Switzerland) and used a single-cone technique as stated by the manufacturer's instructions. Then, a plugger (Cerkamed Medical Company, Poland) was heated to cut off the remaining gutta-percha that exceeded the root canal. Furthermore, the obturation was covered with zinc phosphate cement (Elite Cement 100, GC Corporation Tokyo, Japan) and radiographed to ensure a hermetic obturation system. After that, the sample was conditioned in a plastic container that contained moist gauze for the incubation procedure of seven days at 37°C with 100% humidity.¹⁴

The sample was segmented in the transverse plane, perpendicular to the root canal's long axis using a circular diamond disk (Louyang Penghao Ceramic Technology Co., Ltd, Louyang, China). The apical third of the root was removed with a thickness of 2 mm. The procedure was continued to obtain an apical third sample that would be used in the test. Furthermore, the sectioning was carried out to a thickness of 2 mm and measured with an electronic digital calliper (Mitutoyo, Kawasaki, Japan). The coronal surface of each sample was marked and coded for each group.¹³

The sample was positioned on the surface of a custom-made, cylindrical, resin fixture (20 mm diameter x 7 mm height) with a hole in the middle (2 mm diameter), which would accommodate material dislodgement during the push-out test.¹⁴ The push-out test was conducted with a universal testing machine (TN 20 MD, France) with a 0.53 mm stainless-steel plunger (custom made) that pushed the filling material at a crosshead speed of 1 mm/min in apical-coronal direction.¹⁹ The bond strength was calculated by the following formula:¹³

$$PBS = F / A$$

Where: PBS = push-out bond strength (MPa), F = maximum load (N), A = bonding area of root canal filling (mm²), calculated by the following formula:²⁰

$$A = \pi (r_1 + r_2) \sqrt{(r_1 - r_2)^2 + h^2}$$

Where: $\pi = 3.14$, r_1 = coronal radius, r_2 = apical radius, h = sample thickness.

The data were processed using IBM SPSS Statistics for Windows version 26.0 (IBM Corp., Armonk, NY, USA) for normality with the Shapiro–Wilk test and homogeneity was carried out with Levene’s test. Statistical analysis was conducted with a one-way ANOVA test to compare each group. Furthermore, the data were further tested with a post hoc test using the Bonferroni method to determine the value of comparison between groups (significant level set as $p < 0.05$).

RESULTS

The mean, standard deviation, and one-way ANOVA significant value of filling-material bond strength are described in Table 1. The one-way ANOVA test implied that the bond strength value of the filling material was statistically different among final irrigation solution groups ($p < 0.05$). According to the summary of the Bonferroni test in Table 2, there was a difference that was statistically significant between the 2.5% NaOCl group compared to the 20% citric acid group ($p < 0.05$). Otherwise, the differences between the final irrigation 2.5% NaOCl group compared to the 17% EDTA and between 17% EDTA group compared to 20% citric acid were not statistically significant.

Table 1. One-way ANOVA test result: The push-out bond strength of root canal filling material with final irrigating solution 2.5% NaOCl, 17% EDTA, and 20% citric acid

Final irrigation group	N	Mean ± Standard Deviation	Sig
2.5% NaOCl	7	2.05 ± 0.75 MPa	
17% EDTA	7	2.75 ± 0.60 MPa	0.041*
20% Citric Acid	7	2.98 ± 0.59 MPa	

N: Number of specimens, *: Value shows a significant difference at $p < 0.05$.

Table 2. Post hoc test result using the Bonferroni methods: The push-out bond strength of root canal filling material with final irrigating solution 2.5% NaOCl, 17% EDTA, and 20% citric acid

Final irrigation group	2.5% NaOCl	17% EDTA	20% Citric Acid
2.5% NaOCl		0.182	0.048*
17% EDTA			1.000
20% Citric Acid			

*: Value shows a significant difference at $p < 0.05$.

DISCUSSION

The push-out test provides information about the material bonding properties and material resistance, and it is intended to assess the degree of material bonding to the dentin. As the push-out bond-strength value of the filling material gets higher, the adhesion of the material also gets better. In endodontics, the push-out test is conducted to study filling material resistance, perforation improvement, post retention, and sealer bonding to dentin.^{10,21} Moreover, the push-out test provides better outcomes in assessing the bond strength of intra-canal materials than the conventional shear test method. This is due to the dislodgment of material that occurs parallel to the dentin and thus is more useful in representing the clinical setting.²²

The current study complies with the research of Alkudairy *et al.*²³ and Rocha *et al.*,²⁴ which reported a deleterious effect on the filling material bond strength when 2.5% NaOCl was used as the final irrigation solution. This research confirmed that NaOCl as a single irrigation solution does not effectively eliminate the smear layer. The physicochemical properties of NaOCl only work on the organic portions of the smear layer.²⁵ An NaOCl irrigation solution can degrade dentin collagen. Consequently, it affects sealer bond strength.²⁶ AH Plus sealer is chemically bonded with collagen.²⁷ Collagen is the main component of dentin, which plays a critical role in the bonding between the resin sealer and dentin.²⁸ The bonding mechanism of the epoxy resin sealer is the arrangement of covalent bonds from the open epoxide ring to the amino group of collagen dentin.²⁹ Thus, the removal of collagen fibrils from the dentin due to the use of an NaOCl irrigation solution leads to a decrease in the bond strength value of the adhesive system.²⁸

The deproteinisation effect of NaOCl makes the amino group of the collagen become unstable and easily dissolve.³⁰ This produces a less receptive dentin surface, provides weak micromechanical bonds, and decreases the bond strength of the epoxy resin sealer.^{27,30} The deproteinisation of dentin that is irrigated by NaOCl leads to hydrophilic surface properties that do not support the spreading of the hydrophobic AH Plus sealer.³¹

The removal of dentin organic matrix (fragmentation between carbon atom bonds and degradation of the primary structure of collagen) potentially restrains the hybrid layer formation. After breaking down long collagen chains, NaOCl also chlorinates protein terminal groups. The presence of chloramine protein results in premature termination of the polymer chain and incomplete resin polymerisation.²⁷ The release of oxygen from NaOCl may inhibit the polymerisation process and thereby reduce bond strength, especially in the apical third.^{23,24} Besides, a negative correlation has been found between the exposure time of NaOCl and the material bond strength.²⁶

A combination of NaOCl and the chelating agent has a positive effect on the push-out bond strength of

the epoxy resin sealer.^{6,32} The chelating agent that was used in this study, EDTA 17% (One Med) and 20% citric acid (Biochemistry Laboratory, Universitas Lambung Mangkurat), indicated similar bond-strength values. Filling material that previously was irrigated with 2.5% NaOCl - 17% EDTA and 2.5% NaOCl - 20% citric acid indicated higher bond strength in comparison with the group that was irrigated with 2.5% NaOCl as a single irrigation solution. The current study is in accordance with the research by Berástegui *et al.*²⁵ that showed that a higher bond strength was obtained when NaOCl was combined with a chelating agent. However, statistical analysis of the current study is in line with Farag *et al.*,¹³ who conducted a push-out test of filling material with different irrigation solutions. The study reported that the difference of filling material bond strength between the irrigation group with 2.5% NaOCl compared to the irrigation group with 2.5% NaOCl-17% EDTA was not statistically significant.¹³

Alkudhairi *et al.*²³ and Gündoğar *et al.*³³ explained that smear layer removal became more effective when the root canal was irrigated with EDTA solution and provided the higher bond-strength value than irrigation with an NaOCl solution. Irrigation with EDTA showed the higher bond-strength value was caused by its effectiveness in removing the smear layer, demineralising dentin, opening dentin tubules, and increasing dentin surface roughness.^{23,34} Therefore, 17% EDTA, which was used as the final irrigation solution, facilitated collagen exposure, increased sealer spreading, and established a dentin substrate more conducive to AH Plus adhesion.³⁵

Final irrigation with EDTA shows a higher bond-strength value because EDTA can significantly reduce dentin wetting, thereby resulting in a dentin substrate that has a favourable condition for hydrophobic properties of AH plus.^{5,36} A comparative evaluation of the contact angle suggests that the contact angle of the sealer is reduced after irrigation with EDTA 17%.³⁷ The AH plus sealer shows better surface wetting in the application of EDTA and NaOCl irrigation solutions than using NaOCl irrigation solutions only. This is due to the intimate contact between the dentin surface and the sealer, possibly achieved by adequate smear layer removal, which enhances sealer infiltration into the dentinal tubules.³⁸

The effect of EDTA on dentin is determined by its concentration and time of exposure.³⁹ In the present study, the final irrigation was intended for three minutes as mentioned by Mello *et al.*,¹⁵ who suggested that root canal irrigation with 5 ml of EDTA for three minutes could eliminate the smear layer effectively. Besides, the dynamic manual agitation technique was also used during irrigation. This technique has been proven to be more capable of removing dentinal debris, the smear layer, and biofilm than the static irrigation technique.¹⁷ Statistical analysis of the current study indicated that the bond strength of the final irrigation group EDTA 17% and the final irrigation group 20% were not significantly different. The current study is in line with Ravikumar *et al.*³⁶ who examined the bond

strength of filling material with these chelating agents. The study showed that the difference of bond strength among the final irrigation group with EDTA and citric acid was not significant.³⁶

Different concentrations of citric acid (1–50%) have been widely used in removing the smear layer.⁷ Citric acid with a concentration of 20% was used in the current study because of its biocompatibility and capacity to demineralise the inorganic portion of the smear layer. This was conformable with the study that stated that concentrations of 1–40% citric acid were adequate in eliminating the smear layer, dissolving dentin debris, and demineralising intra-tubular dentin to expose the dentinal tubules. Besides, citric acid with a concentration of 20% does not have any detrimental effect on the dentin surface. Based on this reasoning, the current study was carried out using a concentration of 20% to increase its capacity as a chelating agent.¹⁸ The highest bond strength of the final irrigation group with 20% citric acid can be attributed to the previous study, which showed that the root canals that were irrigated with citric acid showed more effectiveness than the 17% EDTA in eliminating the smear layer at the apical and middle third portion of the root canal. Besides, the application of citric acid with a concentration of 20% also increases the chelating effect.^{12,25}

Several factors that determine the effect of the chelating agent are contact time, pH, concentration, and the volume of the solution. Research conducted by Berástegui *et al.*²⁵ showed a similar capacity for the smear layer removal between 20% citric acid and 17% EDTA. The concentration of citric acid 20% does not have any damaging effect on the surrounding tissue because it is not highly ionised. Another study that applied citric acid at a concentration of 20% showed that the chelating effect of citric acid became higher with increased concentrations. The application of 20% citric acid as a chelating agent results in an increase in the contact area and covalent bond, thereby resulting in a higher AH Plus sealer bond to the dentin.^{25,40}

However, due to its ability to disinfect and dissolve organic tissue, NaOCl irrigation remains an option in contemporary endodontics, although the current study showed the lowest filling material bond strength compared to other groups.^{13,41} NaOCl does not remove the smear layer that coats the dentin and occludes the orifice of the dentinal tubules, thereby restricting the sealer penetration into dentinal tubules.^{13,41,42} Meanwhile, the use of EDTA or citric acid as final irrigation solution can remove the smear layer and open the dentinal tubules, which facilitates the collagen exposure in intratubular dentin, thereby providing a higher filling material bond strength as the adhesion mechanism of the epoxy resin sealer is an arrangement of covalent bonds by any exposed amino groups in dentin collagen to the open epoxide ring of AH Plus sealer.^{29,41}

The bond strength of the epoxy resin sealer is also associated with creep capacity, low shrinkage levels during setting, flow-ability properties, low polymerisation shrinkage, sealer volumetric expansion, and long-term

dimensional stability.^{1,2,3} Several studies have found diversity on the sealer bond strength values when the samples were examined with different root canal depth-level sections.^{13,19,23,29} Nonetheless, the current research used samples in the apical one-third that tended to show the lowest value of bond strength compared to the middle and coronal third.³ The reduction in root canal diameter, anatomic variation, and vapour lock effect in the apical third interferes with the irrigation solution flow and makes removing the smear layer even more challenging.¹² The decrease in dentinal tubule density, sclerotic dentin, and inhomogeneous hybridisation of dentin in the apical third also reduces the level of material adhesion to dentin.^{3,6,23,43} The results of the current study indicate that there are differences in the bond strength of root canal filling material with different final irrigation solutions. The push-out bond strength of root canal filling material is increased by applying a chelating agent as final irrigation. The final irrigation with 20% citric acid shows the highest bond strength value of filling material and implies a significant difference in bond strength compared to 2.5% NaOCl.

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