

Effectiveness of Three Different Application Times of 17% EDTA and 7% Maleic Acid Irrigation Agents on the Removal of Debris and Smear Layer: A Scanning Electron Microscope Study

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Abstract

Aim: This study compared smear-layer and debris removal using with three different application times of 17% EDTA and 7% maleic acid irrigation agents.

Methodology: Fifty-two maxillary and mandibular single-rooted teeth were divided randomly into seven groups (n = 8 each, control group n = 4). Three groups received 17% EDTA and three received maleic acid (1, 5, 10 min). Canals were prepared using nickel-titanium rotary files. Smear-layer and debris removal were evaluated in the coronal, middle, and apical thirds of the teeth using scanning electron microscopy, and results were analyzed compared using Mann–Whitney U tests.

Results: Maleic acid removed the smear layer (coronal and middle: $p < 0.01$; apical: $p < 0.05$) and debris (all $p < 0.05$) more effectively than EDTA in terms of 1 min application period. Effectiveness did not differ significantly with 10-min applications.

Conclusions: Final irrigation with 7% maleic acid is more efficient than 17% EDTA in the removal of smear layer and debris from the coronal, middle and apical third of the root canal system in application within 1 min.

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Key Words

EDTA, maleic acid, smear layer, debris.

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Introduction

One of the major advantages of root canal treatment is the achievement of clean canals before filling (1). Micro-organisms can be controlled in infected root canals through adequate biomechanical expansion of the canal and effective canal irrigation. It is also extremely important that organic and inorganic residues and debris are removed from canals effectively (1,2).

Microscopic evaluation has shown that root canals vary considering the irregular and complex secondary canals. Carious and traumatic lesions may also change the character of the tooth pulp and allow the invasion of pathogens and toxins into dentin canals. Such invasions devitalize the pulp, negatively impacting the ability to treat the canal successfully (3). The success of root canal treatment depends on good shaping, removal of infected tissues, and proper filling of the canal. Previous

studies have shown that the instruments used in canal expansion and shaping generate a smear layer that covers the dentine walls (4). The smear layer, which contains bacterial cells, impedes proper canal filling and prevents the diffusion of dentine canals (1). It has been shown that the smear layer itself may be infected and may protect the bacteria within the root canal dentinal tubules (1). Various chelating agents like citric acid, EDTA, and a mixture of tetracycline isomer (doxycycline), an acid (citric acid) and a detergent (Tween 80) have been used for the removal of the smear layer in root canal walls (5,6). The use of a combination of sodium hypochlorite (NaOCl) and the chelating agent EDTA has been recommended for effective removal of the smear layer (6). Particularly some researchers have suggested the use of 17% ethylenediaminetetraacetic acid (EDTA) and 5% sodium hypochlorite (NaOCl) for the effective cleaning of root canals (7,8). However, some researchers have also been reported following the combined use of these materials, such as the erosion of dentine walls (9-11).

The strong demineralising effect of EDTA can cause enlargement of dentine tubules, softening of dentine, and denaturing of collagen fibres. These effects can impede the adaptation of canal filling materials to dentine walls (12). Bystrom et al. (13) determined that 5% NaOCl with 15% EDTA removed bacteria and the smear layer more effectively than 5% NaOCl alone. Although NaOCl removes organic components effectively, it has been reported to have no impact on the removal of inorganic components. It should thus be used in combination with chelating agents to ensure proper root canal debridement (6,7,14).

Maleic acid is a mild organic acid used as an acid conditioner in adhesive dentistry (15). It has been found to acquire the smear layer-removing high quality when used as an acid etchant in restorative dentistry (15,16). So far, there was only a few study published evaluating the different concentrations (5%, 7%, 10%, or 15%) of maleic acid in the removal of the smear layer from the root canal dentin (17,18).

The purpose of this study was to conduct a scanning electron microscopic (SEM) analysis comparing the effectiveness of 1-, 5-, and 10-min applications of NaOCl / 17% EDTA and NaOCl / 7% maleic acid in the removal of the smear layer and debris in the coronal, middle, and apical thirds of root canals.

Materials and Methods

The study sample consisted of 52 extracted maxillary and mandibular single-rooted non-carious teeth, chosed randomly from male and female

patients between the ages of 20 and 45 years. The teeth were soaked in 0.5% chloramine-T solution for 1 week to prevent bacterial growth. Before preparation, all teeth were cleaned with distilled water. The crowns were removed at the cemento-dentinal junction with a diamond disk (KG Sorensen, Barueri, SP, Brazil). Working lengths were defined 1 mm short of the apices with a #10 K-file. Using the crown-down technique, the root canals were prepared to size 30 (F3, 0.04) with ProTaper nickel-titanium (Ni-Ti) rotary instruments (Dentsply-Maillefer, Ballaigues, Switzerland). Each instrument was replaced after it had been used on two teeth. An irrigation agent (5 mL 2.5% NaOCl solution) was applied with a 27-gauge needle during each rasping procedure. After instrumentation and irrigation, the teeth were randomly divided into seven groups for the final irrigation regime. All of the solutions used were prepared freshly. The groups received the following treatments:

Group A (n = 8): 5 mL 17% EDTA (Roth International Ltd., Chicago, IL, USA) for 1 min, followed by 5 mL distilled water;

Group B (n = 8): 5 mL maleic acid (KMC Pharmacy, Karnataka, India) for 1 min, followed by 5 mL distilled water;

Group C (n = 8): 5 mL 17% EDTA for 5 min, followed by 5 mL distilled water;

Group D (n = 8): 5 mL maleic acid for 5 min, followed by 5 mL distilled water;

Group E (n = 8): 5 mL 17% EDTA for 10 min, followed by 5 mL distilled water;

Group F (n = 8): 5 mL maleic acid for 10 min, followed by 5 mL distilled water;

Group G (control, n = 4): 5 mL 2.5% NaOCl, followed by 5 mL distilled water.

Thereafter, all teeth were dried with paper points.

SEM Evaluation

For SEM examination, the buccal and lingual surfaces of all roots were grooved longitudinally with a fissure diamond bur in a high-speed handpiece (diameter 125 mm X 0.35 mm X 12.7 mm), under copious water irrigation, avoiding penetration of the root canals. The specimens were then placed in a desiccator for at least 48 h. The samples were bisected and both halves were mounted in specimen holders. They were then placed in a vacuum chamber and coated with a 25- μ m thick gold-palladium layer for SEM (JEOL T330; JSM, Tokyo, Japan) analysis. After the samples were coated with 10 μ m of gold, SEM photomicrographs were taken at 2500 \times magnification. The coronal, middle, and apical thirds of all samples were evaluated.

Image analysis

Two operators evaluated the SEM images using a double-blind method and the scoring system of Ahmad et al. (19) The debris and smear-layer quantities were each given a score between 0 and 3, as follows:

Debris scores:

- 0 = no superficial debris on the dentine wall;
- 1 = minimal debris;
- 2 = moderate debris;
- 3 = heavy amounts of debris.

Smear scores:

- 0 = no smear on the dentine wall, all tubules opened;
- 1 = light smear, >50% of tubules opened;
- 2 = moderate smear, <50% of tubules opened;
- 3 = heavy smear, outlines of tubules obliterated.

Intra- and interexaminer reliability for the SEM assessment was verified by the Kappa test. Mean debris and smear-layer scores were calculated for each tooth and group, and the groups were compared using the Mann–Whitney U test at a significance of .05. The results obtained at each evaluation level within each group were analysed using Friedman's nonparametric two-way analysis of variance (ANOVA). Statistical data were evaluated using the SPSS software (ver. 10.0; SPSS Inc., Chicago, IL, USA).

Results

Table 1 presents the results of different application time of EDTA and maleic acid solutions on removal of the smear layer and debris in the coronal, middle, and apical thirds of the root canals.

TABLE 1. P values for smear layer removal among groups A to F

Canal Level	Time	Group Comparison	p Value
Coronal	1 min	Group B was cleaner than group A	p<0.01
Coronal	5 min	No significant difference between groups C and D	p=0.264
Coronal	10 min	No significant difference between groups E and F	p=0.293
Middle	1 min	Group B was cleaner than group A	p<0.01
Middle	5 min	No significant difference between groups C and D	p=0.175
Middle	10 min	No significant difference between groups E and F	p=0.530
Apical	1 min	Group B was cleaner than group A	p<0.05
Apical	5 min	Group D was cleaner than group C	p<0.01
Apical	10 min	No significant difference between groups E and F	p=0.165

TABLE 2. P values for debris removal among groups A to F

Canal Level	Time	Group Comparison	p Value
Coronal	1 min	Group B was cleaner than group A	p<0.05
Coronal	5 min	No significant difference between groups C and D	p=0.589
Coronal	10 min	No significant difference between groups E and F	p=0.424
Middle	1 min	Group B was cleaner than group A	p<0.05
Middle	5 min	No significant difference between groups C and D	p=0.114
Middle	10 min	No significant difference between groups E and F	p=0.575
Apical	1 min	Group B was cleaner than group A	p<0.05
Apical	5 min	Group D was cleaner than group C	p<0.05
Apical	10 min	No significant difference between groups E and F	p=0.069

Smear layer removal

Kappa test results, with a significance set at .05 and .01 (1 and 5 min.), showed good intra- and interexaminer agreement. After 1-min applications, the amount of smear layer removal differed significantly in the coronal, middle (both $p < 0.01$), and apical ($p < 0.05$) thirds of the root canals. After 5-min applications, smear layer removal differed

significantly only in the apical third ($p < 0.01$). No significant difference was observed after 10-min applications. maleic acid removed the smear layer more effectively than EDTA (Fig. 1), especially with an application of short duration (1 min).

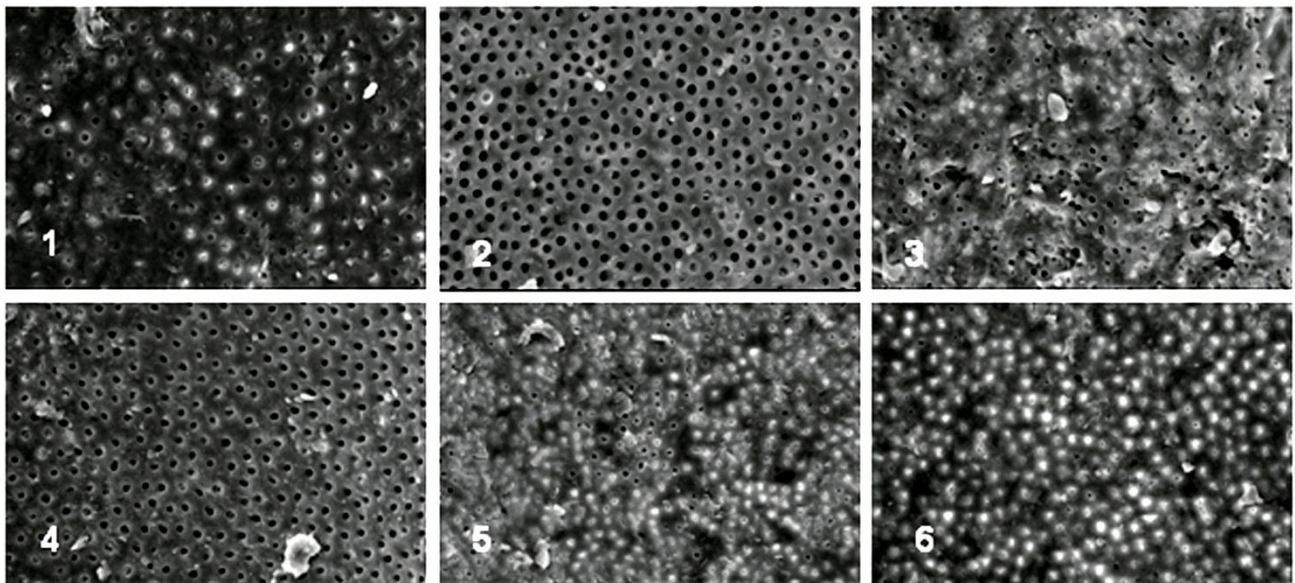


Figure 1. Scanning electron microscopic images of root canal walls (2500 \times). **(1)** Group A, EDTA (1 min), coronal third (smear score 2, debris score 1); **(2)** Group B, maleic acid (1 min), coronal third (smear score 0, debris score 0); **(3)** Group C, EDTA (5 min), middle third (smear score 2, debris score 1); **(4)** Group D, maleic acid (5 min), middle third (smear score 0, debris score 0); **(5)** Group E, EDTA (10 min), apical third (smear score 3, debris score 2); **(6)** Group F, maleic acid (10 min), apical third (smear score 3, debris score 2).

Debris removal

Kappa test results, with a significance set at .05 (1 and 5 min.), showed good intra- and interexaminer agreement. After 1-min applications, the amount of debris removal differed significantly in the coronal, middle, and apical (all $p < 0.05$) thirds of the root canals. After 5-min applications, debris removal differed significantly only in the apical third ($p < 0.05$). No significant difference was observed after 10-min applications. maleic acid removed debris more effectively than EDTA (Fig. 1), especially with an application of short duration (1 min).

The control group demonstrated that NaOCl was ineffective for the removal of the smear layer and debris (Fig. 2).

Table 2 presents the results of intra-group evaluations, conducted using Friedman's ANOVA. In the middle third of the root canal, EDTA removal of the smear layer differed significantly with application

length ($p < 0.05$). No significant difference was observed for the removal of smear layer and debris in the coronal and apical thirds with different lengths of EDTA application. Maleic acid intra-group comparison revealed no significant difference in the removal of smear layer and debris among application lengths and root canal portions.

Discussion

The purpose of this study was to evaluate the smear-layer and debris removal effects of three application lengths of 7% maleic acid and 17% EDTA during final irrigation, following initial irrigation with 2.5% NaOCl.

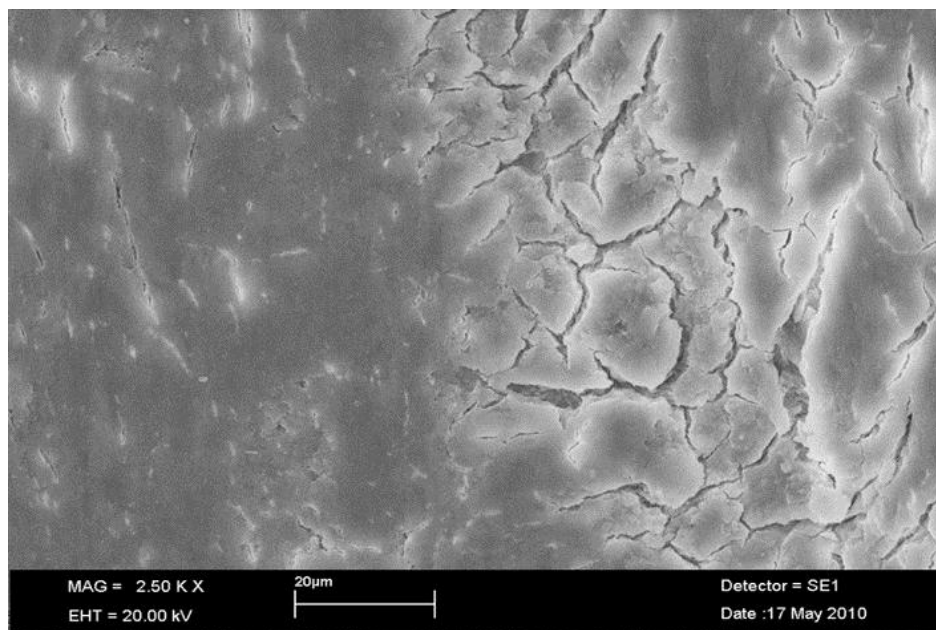


Figure 2. Scanning electron microscopic images of root canal walls (2500×). Control group, NaOCl (smear score 3, debris score 3).

Many researchers have demonstrated that debris and the smear layer occur during canal expansion and shaping as a result of the mechanical expansion applied to root canals (20,21). The debris and smear layer provide a favourable environment for the reproduction of microorganisms and bacteria, and may distort the adhesion of root canal fillings to the canal wall. This distortion may, in turn, lead to apical leakage, preventing the expansion of intra-canal medicaments into dentine canals (1,2). Components that will negatively impact the success of canal treatment must be removed from root canals; several tissue solvents and removal solutions are used for this purpose during endodontic treatment. Irrigation solutions must dissolve organic and inorganic components and remove the smear layer. The widely used NaOCl irrigation agent has superior tissue-solvent characteristics, but is insufficient for the removal of inorganic components. Researchers have thus sought other agents, such as EDTA (11), MTAD (6), maleic acid (17,18), and citric acid (7) chelating agents. These agents remove inorganic components, debris, and the smear layer effectively when used in combination with NaOCl (3,11,22).

EDTA is a widely used chelating agent in endodontic treatments (23). Many studies have reported that the combined use of EDTA and NaOCl removes the smear layer effectively; the most effective final irrigation has been reported to be 10 mL 17% EDTA, followed by 10 mL 5.25% NaOCl (7,8,24).

O'Connell et al. (25) reported that the combined use of EDTA and 5.25% NaOCl for canal irrigation removed the smear layer effectively in the middle and coronal thirds of the root canal, but achieved only inefficient removal in the apical third. They suggested that this shortcoming was due to insufficient irrigant volume or insufficient penetration into the apical third of the canal. The results of the present study were consistent with their findings.

Çalt and Serper (9) compared 1-min and 10-min applications of 17% EDTA to root canals as an irrigation agent. The smear layer was removed effectively with a 1-min application, whereas peri- and intra-tubular erosion occurred during a 10-min application. Gettleman et al. (26) showed that a contact time of 3 min with 17% EDTA was effective for smear layer removal.

Ballal et al. (18) reported that 7% maleic acid followed by 2.5% sodium hypochlorite had a better smear layer removal ability than 17% EDTA followed by 2.5% sodium hypochlorite in the apical third of the root canal system during the application for 1 min. In the middle and coronal third, both EDTA and maleic acid were equally effective without any statistical difference between them. The results of our study for 5 min. (smear layer and debris) are consistent with their findings. However, application for 10 min. (all regions) showed no significant difference between EDTA and maleic acid groups. Furthermore, in the present study maleic acid had a better smear layer and debris removal ability than 17% EDTA in the coronal, middle, and apical thirds of the root canal system during the application for 1 min.

EDTA did not remove smear layer and debris effectively when compared with maleic acid in root canal walls. This maybe due to the increased surface tension of 17% EDTA (0.0783 N/m) when compared with that of 7% maleic acid (0.06345 N/m) (18). Since EDTA is a chelating agent, it is effective at a neutral pH and it is independent on a high hydrogen ion concentration to induce decalcification. Calcium released from dentin decreases pH with the resulting hydrogen. With a positive correlation the efficacy of EDTA decreases over time because of the decrease in pH (2). Since maleic acid is highly acidic, it demineralises within a shorter period of time. However, EDTA requires an application time of not less than 15 minutes for optimal results (27). EDTA solution reduces the mineral and noncollagenous proteins of dentin (2). As Hülsmann et al. (2) reported that the content of noncollagenous proteins component decreased in the apical third of the root canal system, the degree of decalcification of EDTA in this part was low. Also, Çalt et al. (9) and Niu et al. (10) reported that when was EDTA used for more than 1 minute it had caused erosion of dentinal tubules. Kuah et al. (28) compared the effectiveness of 1-min and 3-min ultrasonic applications of EDTA on smear layer removal, finding no significant difference. They reported that a 1-min application removed the smear layer in the tested region. Jiang et al. (29) applied irrigation agents with sonic tools and evaluated debris removal in root canal thirds. They found that this application improved the activation of irrigants and removed debris effectively. Mello et al. (30) reported that a 3-min application of 3 mL 17% EDTA as a final solution effectively removed the smear layer in root canals. NaOCl, which is used as an irrigant in the control group, was found to have no effect on the smear layer and debris removal.

Conclusion

Within the limitations of this study, a 1-min application of 7% maleic acid during final irrigation removed the smear layer and debris more effectively than a 1-min application of 17% EDTA in the coronal, middle, and apical thirds of root canals.

Acknowledgments

The authors deny any conflicts of interest related to this study.

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