Effect of different irrigating solutions with surfactants on the microhardness and smear layer removal of root canal dentin: An *in vitro* study

Rajan Dhawan, Ankit Gupta, Jaidev Singh Dhillon, Shivani Dhawan, Tamanna Sharma, Divya Batra

Departments of Conservative Dentistry and Endodontics and *Periodontics, Maharishi Markandeshwar College of Dental Sciences and Research, M.M.U Mullana, Ambala, 1Department of Conservative Dentistry and Endodontics, Swami Devi Dyal Hospital and Dental College, Barwala, Haryana, 2Private Practice, Delhi, 3Department of Conservative Dentistry and Endodontics National Dental College and Hospital Derabassi, Punjab, India

**Abstract**

**Aim:** The present *in vitro* study was undertaken to check the effect of the different irrigating solutions with surfactants, i.e., sodium hypochlorite-(Naocl)-Extra, chlorhexidine (CHX)-Ultra, ethylenediaminetetraacetic acid (EDTA), QMix, and BioPure MTAD on the microhardness and smear layer removal of root canal dentin.

**Materials and Methods:** A total of 120 straight rooted lower premolars were collected and were randomly divided into 2 equal groups of 60 each (*n* = 60). The microhardness of the samples was evaluated by Vickers hardness tester and the removal of smear layer by scanning electron microscope after irrigation of the samples with the tested solutions.

**Results:** CHX-Ultra showed the least microhardness reduction, and EDTA showed the maximum microhardness reduction in all the tested groups. BioPure MTAD showed the maximum removal of smear layer in the apical third, and CHX-Ultra showed the minimal smear layer removal in the apical third.

**Conclusion:** During smear layer removal, irrigating solutions cause alterations in the chemical composition of dentin, which may decrease the microhardness of the root dentin causing erosion and affecting the clinical performance of the endodontically treated teeth. Irrigating solution with maximum smear layer removal with minimum changes in microhardness should be used.

**Keywords:** Irrigants; microhardness; smear layer

**INTRODUCTION**

Irrigation is currently the best method for the removal of loose, necrotic, and infected pulp tissue remnants during root canal treatment before they are pushed deeper into the canal and the apical tissues. The success rate of endodontic therapy increases as more debris and smear layer are removed.[1]

Smear layer is a porous layer that reduces the penetration of irrigating solutions and intracanal medicaments into the root canal system and dentinal tubules. It also prevents proper adaptation of obturation materials to the prepared canal surfaces. Irrigants prevent the formation of smear layer during instrumentation or dissolve it when it forms.[2]

None of these irrigants can easily reach target areas within the intricate structure of the root canal system. To achieve deeper accessibility of irrigants into the dentinal tubules and lateral canals, surface-active agents (surfactants) are being added that reduce the surface tension of the irrigant and act as detergents, wetting agents, emulsifiers, foaming agents, or dispersants.[3]

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Sodium hypochlorite (NaOCl)\(^1\) is the most widely used irrigating solution with excellent antimicrobial action, but it does not effectively remove the smear layer. NaOCl-Extra contains 6% NaOCl and surface modifiers with improved penetrative ability.

EDTA effectively removes smear layer and dentin mud and opens dentin tubules for disinfection solutions to work better and for the better adhesion of sealers and obturation materials.

Chlorhexidine (CHX)-Ultra kills multispecies biofilm two times faster and planktonic bacteria ten times faster than standard 2% CHX, and surface modifiers enable penetration into hard-to-reach areas such as lateral canals and isthmuses.

BioPure MTAD (Dentsply) had been shown to effectively eliminate smear layer and Enterococcus faecalis that are resistant to conventional endodontic irrigants.\(^4\)

QMix (Dentsply) is a novel endodontic irrigant with antimicrobial agents that can efficiently remove smear layer. It consists of EDTA, CHX, and detergent.\(^5\)

However, with smear layer removal, irrigating solutions cause alterations in the mineral content of dentin, which may decrease its microhardness, that affect the sealing ability and the adhesion of resin-based cement and root canal sealers to dentin.\(^6\) Microhardness is regarded as indirect evidence of changes of mineral content in root canal dentin.\(^7\)

This in vitro study evaluated the effect of different root canal irrigants with surfactant on dentin microhardness and smear layer removal in the root dentin.

**MATERIALS AND METHODS**

The present study was approved by the institutional ethical committee. Freshly extracted 120 straight single-rooted lower premolars with almost similar dimension and morphology were taken. The presence of single canal and the absence of any caries, cracks, curved canals, previous endodontic treatment, internal resorption, or calcification of canals were confirmed by intra oral periapical radiograph.

Teeth were cleaned from soft and/or hard attached tissues, decontaminated by immersion in 5.25% NaOCl solution for 30 min, sterilized as per the Centers for Disease Control and Prevention, and were stored in sterile saline solution at room temperature throughout the study period. The crowns of all teeth were cut transversally at the cementoenamel junction with double-faced diamond disc at slow speed, along with water coolant, to get 15 mm ± 0.5 mm root length.

The working length of all teeth was obtained by measuring the length with #10 K-file at the apical foramen minus 1 mm. The canals were enlarged till #15 K-file to establish glide pathway. ProTaper rotary instruments were used for biomechanical preparation. Instruments were used till apical size F3.

Specimens were randomly divided into two equal parts of sixty each. The first part \((n = 60)\) was named as Group M to test the surface microhardness of root canal dentin, and the second part \((n = 60)\) was named as Group S to observe smear layer by scanning electron microscope (SEM).

**Microhardness evaluation**

For microhardness evaluation of each group, the longitudinal sectioning of the root was done, and longitudinal grooves were made on buccal and lingual external root surfaces by double-faced diamond disc at slow speed as not to penetrate the root canals. The specimens were then splitted into two segments by chisel, thus getting one twenty halves. Each root half was horizontally embedded in acrylic block and was assigned a private number. Testing was carried out using Vickers microhardness tester at baseline and after the use of irrigating solution.

Specimens were divided randomly into six equal groups, according to the irrigant to be used, with twenty samples in each group:

- **Group M1**: NaOCl-Extra (Coltene Endo)
- **Group M2**: Pro-EDTA (Coltene Endo)
- **Group M3**: CHX-Ultra (Coltene Endo)
- **Group M4**: MTAD (Dentsply)
- **Group M5**: QMix (Dentsply)
- **Group M6** (control group): 5 ml normal saline.

Specimens were kept in respective irrigant solutions for 5 min in closed glass plates at 37°C. Flushing of specimens was done with 30 ml sterile saline and then dried with sterile paper points. The posttreatment microhardness was then measured.

**Part II: Smear layer evaluation**

Sixty teeth were taken and grouped as:

- **Group S1**: NaOCl-Extra (Coltene Endo)
- **Group S2**: Pro-EDTA (Coltene Endo)
- **Group S3**: CHX-Ultra (Coltene Endo)
- **Group S4**: MTAD (Dentsply)
- **Group S5**: QMix (Dentsply)
- **Group S6** (control group): 5 ml normal saline.

Each group was initially irrigated with 1 ml of the experimental solution. A 30G needle, which penetrated to within 1–2 mm from the apex, was used for initial and final irrigation. To ensure a uniform and direct contact of each irrigant with the root canal walls, a #15 barbed broach was wrapped with
cotton and soaked with one of the experimental solutions and placed to the working length. After 4 min, the wrapped broach was moved up and down 4–5 times, and then, each group was irrigated with 4 ml of respective experimental solution as a final rinse. The total exposure time to the final solution was approximately 5 min.

The canals were then irrigated with 10 ml of sterile distilled water, dried by paper points and orifices were sealed with a small cotton pellet to avoid contamination of canal space during sectioning procedure. In the control group, the canal was only irrigated with 5 ml normal saline and dried by sterile paper points.

Two longitudinal grooves were made on the palatal/lingual and buccal surfaces of each root with diamond disc. Each root was splitted longitudinally into two halves with chisel, getting twenty root halves for each group.

**Scanning electron microscope preparation**

The roots sections were numbered, mounted on metallic stubs, gold sputtered to make the surface electrically conductive, and evaluated by SEM in the apical one-third of root dentin.

The amount of smear layer left on the root canal surface or in the dentinal tubules was calculated as:

- **Score 1:** No smear layer, dentinal tubule orifice completely patent
- **Score 2:** <25% of canal area covered by a thin smear layer with visible dentinal tubule opening
- **Score 3:** Patchy distribution of smear layer covering up to 50% of canal wall
- **Score 4:** Thin smear layer uniformly covering the entire canal wall
- **Score 5:** Thick smear layer uniformly covering the entire canal wall.

**RESULTS**

Statistical analysis for microhardness was performed using means of one-way ANOVA, *post hoc* tests for intergroup comparison. For smear layer removal, one-way ANOVA, Kruskal–Wallis tests were used for intergroup comparison.

According to the mean differences between the different groups, the results of change in microhardness were [Graph 1]:

- EDTA > BioPure MTAD > QMix > NaOCl-Extra > CHX-Ultra > saline.

The minimum microhardness change was seen in the CHX-Ultra group and the maximum in EDTA. The mean difference in microhardness proved statistically significant (*P* ≤ 0.05).
The results of smear layer removal were [Figures 1-6]:

**Figure 3:** Scanning electron microscope showing the effect of BioPure MTAD on the apical third

**Figure 4:** Scanning electron microscope showing the effect of BioPure MTAD on the apical third

**Figure 5:** Scanning electron microscope showing the effect of QMix on the apical third

**Figure 6:** Scanning electron microscope showing the effect of the control group (saline) in the apical third

The results of smear layer removal were [Figures 1-6]:

BioPure MTAD > EDTA > QMix > NaOCl-Extra > CHX-Ultra > saline.

The maximum smear layer was with the saline group as compared with the test irrigants. The mean difference in smear layer removal was also proved statistically significant.

**DISCUSSION**

Irrigation by syringe can control both the amount and the depth of penetration of the irrigating solution to the apical region of the root canals. The irrigation in this study was done using the 30G needle as thinner needles (30 gauge) can easily reach the apical area.

Single-rooted lower premolar teeth with single and straight canals were used as they are the most commonly used extracted teeth for orthodontic correction.

The root canals were prepared by passive step-back technique, followed by rotary nickel–titanium instruments. The rotary files generate substantial amount of smear layer. The apical portion of each sample was enlarged to F3 for better penetration of irrigants.

SEM with magnification of ×1000 had been used to evaluate the efficacy of the various irrigants in the removal of smear layer as it allows the evaluation of the morphological details of the surfaces of both halves of canal walls along their complete length.

In the present study, the final irrigation time was 5 min as Lopes et al. reported that in 5 min, a lentulo spiral can completely remove the smear layer from the apical third.

MTAD is a mixture of 3% doxycycline, 4.25% citric acid, and detergent (Tween 80). Doxycycline is bacteriostatic, and Tween 80 increases its antibacterial effect. The results
showed that in Group S4 with MTAD, the apical third showed complete smear layer removal in all specimens.

MTAD has a low surface tension (34.5 mJ/m²) that leads to improve the intimate contact of irrigants with the dentinal walls, permitting deeper penetration for the effective removal of smear layer.[13]

The effect of MTAD on decreasing the dentin microhardness may be attributed to doxycycline (calcium chelator) and citric acid causing demineralization of the root surface.[9]

EDTA is a 17% solution (pH 7) that effectively removes the smear layer by chelating the inorganic components of the dentin, but this study showed only moderate smear layer removal in the apical third. This may be attributed to incomplete penetration of EDTA in apical areas.[14]

Furthermore, EDTA caused the greatest reduction in root dentin microhardness by removing calcium ions and noncollagenous proteins of dentin. The results are in agreement with Singh S et al.[15] that showed a greater reduction in microhardness by EDTA than MTAD; Torabinejad M et al.[9] exhibited more effective removal of smear layer and less decrease in microhardness by MTAD than EDTA in the apical area.

The removal of smear layer by QMix had been shown to be as effective as 17% EDTA on the basis of the number of fully opened dentinal tubules.[3] A unique advantage of CHX is that its ability to adsorb on dentin and prevent microbial colonization. QMix results in reduction of the microhardness of dentin.

The inefficiency of NaOCl-Extra to remove smear layer from the apical third of canals may be attributed to its less physicochemical action restricted to organic particles, which is consistent with the study of Mancini et al.[16] NaOCl-Extra reduces the microhardness as it dissolves both collagen components of dentin and magnesium and phosphate ions, although increasing the volume of dentinal carbonate.[12]

CHX-Ultra was not able to remove the smear layer in the apical third, with minimum effect on the microhardness, and this finding is similar to the studies of Naenni et al.[17] and Ari et al.[18]

Thus, along with the removal of smear layer, these irrigants decrease dentinal hardness indicating their direct effects on dentin, thus weakening the tooth structure.

**CONCLUSION**

It has been concluded that irrigants that remove more of smear layer showed more changes in the microhardness of dentin. The reason may be attributed to the fact that smear layer may act as a barrier that limits the contact of irrigants with dentin, thus allowing minimal changes in its microhardness.[11] Thus, choice of an irrigant should be such that it can efficiently remove smear layer, act as a lubricant during instrumentation, and at the same time, cause minimal changes in the microhardness of root dentin.

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**Conflicts of interest**

There are no conflicts of interest.

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