# Wettability of endodontic sealers on the root surface using different irrigants

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**• Conflicts of interest: none declared.**

## Abstract

**Objective:** the present study aimed to evaluate the wettability of different sealers in contact with dentin treated with irrigants used for smear layer removal, after the use of chlorhexidine as chemical auxiliary substance. **Material and Methods:** thirty single-rooted teeth were used. The crowns were removed and the roots sectioned vertically, resulting in two segments, which were then flattened. Thus, 120 samples measuring 5 mm were obtained. They were immersed in 2% chlorhexidine gel (CHX) for 10 minutes and washed with distilled water (AD) before being subjected to the final irrigant for smear layer removal. The following irrigation protocols were evaluated: control group-CHX+AD+AD+AD; group E-CHX+AD+17%EDTA+AD; group Q-CHX+AD+QMix+AD; group Et-CHX+AD+18%HEBP+AD. The samples were dried and the contact angle was analyzed using a goniometer. The sealers evaluated were: AH Plus (AH), Pulp Canal Sealer EWT (PCS) and MTA Fillapex (MTAF). In each drop of sealer, 60 measurements were made and the flow was observed for 1 minute. The dynamic wettability of the sealers was calculated. Data were statistically analyzed. **Results:** in all sealers, the presence of smear layer disfavored the wettability. EDTA and QMix favored the wettability of AH and PCS. For the MTAF, QMix showed the best performance, followed by EDTA. HEBP showed statistically lower results, similar to the control. **Conclusion:** Solutions used for smear layer removal, after the use of chlorhexidine during preparation, influenced the wettability of AH Plus, Pulp Canal Sealer EWT and MTA Fillapex sealers.

**Keywords:** Flow; Irrigation; Sealers; Smear layer; Wettability.

## Introduction

Root canal therapy is intended to eliminate and prevent infection and re-infection by microorganisms in the root canal system. Chemical substances are used during the preparation to assist disinfection, either by chemical action or physical action.[1]

Chlorhexidine (CHX) has been used as an alternative to NaOCl during chemomechanical preparation, since among other properties it has broad antimicrobial activity, substantivity, lubricating properties, rheological action and chemical stability, besides inhibiting metalloproteinases.[2]

Because CHX does not remove the smear layer, chelating substances and acids are used for this removal.[3] This layer forms a barrier that reduces the permeability of dentin, in turn reducing the access of irrigants and intracanal drugs into dentinal tubules and the bonding strength of filling materials to dentin, favoring coronary microleakage.[3,4] The most common solution used for this purpose is EDTA. Other solutions, like QMix and etidronate, have being proposed to remove the smear layer.[5-7]

QMix is an irrigant with antimicrobial properties consisting of an acid chelating agent, a bisbiguanide anti-microbial agent, a surfactant and deionized water. Its properties have been tested in several recent works, with observance of significant results in terms of smear layer removal when compared to EDTA.[5,6]

1-hydroxyethylidene-1, 1-bisphosphonate (HEBP), also known as etidronic acid or etidronate, is a chelating solution that has been proposed as an alternative to EDTA or citric acid because it is reactive with NaOCl, not toxic, is less aggressive to dentin and does not interfere in increased bond strength.[5,6]

The physicochemical properties of a sealer can characterize its clinical behavior during and after filling. Different analytic techniques have been used to evaluate the adhesion properties of endodontic sealers to dentin treated with different irrigants. The contact angle analysis technique is used to measure the angle between a drop of liquid and the flat surface of the solid. The surface that presents the lowest contact angle will have highest wettability, i.e., higher sealer flow can be observed.[8-10]

The aim of this study was to evaluate the wettability of different sealers in contact with dentin treated with irrigants used for smear layer removal, after the use of chlorhexidine as chemical auxiliary substance. The null hypotheses evaluated were: The use of EDTA (i) QMix (i) or etidronate (iii) after the 2% chlorhexidine gel does not interfere in the dynamic wettability of AH Plus, Pulp Canal Sealer EWT and MTA Fillapex; and the chemical agents EDTA, QMix and etidronate are equally effective (iv).

## Material and Methods

### Sample Preparation

This study was approved by the Ethics Committee on Human Research of Federal University of Rio de Janeiro (Protocol number 934.497). Thirty single-rooted permanent human teeth, with straight roots, apexes with full formation and single canals were used. The crowns were removed at

the level of cemento-enamel joint with a double-sided flexible disk diamond (KG Sorensen Ind. Com. Ltda., Barueri, SP, Brazil) connected to an electric micromotor. Then the teeth were cleaved in the direction of the long axis, resulting in two segments, buccal and palatal. The dentin surface was flattened with a sanding machine (sanding paper granulation number 100, 3M Sumaré, SP, Brazil / Politriz, Vargem Grande Paulista, SP, Brazil) under cooling with water to create a standard smear layer. Sixty flat dentin surfaces were obtained and each surface originated two samples of 5 mm, measured using a digital caliper (Mitutoyo Sul Americana Copyright ©, São Paulo, SP, Brazil), for a total of 120 samples.

**Irrigation Protocols**

Samples were distributed in groups, according to the irrigation protocol used:

- Control Group (CG) – 2% chlorhexidine gel + intermediate flush with distilled water + removal of the smear layer with distilled water + final flush with distilled water;
- Group E (GE) – 2% chlorhexidine gel + intermediate flush with distilled water + removal of the smear layer with 17% EDTA + final flush with distilled water;
- Group Et (GEt) – 2% chlorhexidine gel + intermediate flush with distilled water + removal of the smear layer with 18% etidronate + final flush with distilled water;
- Group Q (GQ) – 2% chlorhexidine gel + intermediate flush with distilled water + removal of the smear layer with QMix + final flush with distilled water.

The samples were immersed in 2% chlorhexidine gel (CHX) (Drogal, Piracicaba, SP, Brazil) during 10 minutes, to simulate the contact time with this substance during the chemical-mechanical preparation. Then they were washed with 5 ml of distilled water to remove traces of CHX, using a syringe. For smear layer removal, 17% EDTA (Maquira Indústria de Produtos Odontológicos Ltda, Maringá, PR, Brasil), Q Mix™ 2 in 1 (Dentsply, Tulsa Dental Specialties, Tulsa, OK, USA)- and 18% etidronate (Zschimmer & Schwarz Mohsdorf GmbH & Co KG, Burgstädt, SN, Germany) were used. The samples were irrigated with 3 ml of solution for 3 minutes (1 mL per minute). Then, a new irrigation with 5 mL of distilled water was performed to remove the traces of irrigant employed for smear layer removal. Finally, the samples were dried with compressed air.

**Scanning Electron Microscopy Analysis**

In each group, three samples were chosen randomly and evaluated by scanning electron microscopy to visualize the degree of smear layer removal promoted by the different irrigant agents.

For this purpose, a JSM 6460 LV scanning electron microscope (JEOL, Tokyo, Japan) was used in environmental mode. In each sample, four microphotographs were obtained at 1000x magnification (12 microphotographs per group).

**Measurement of the Contact Angle Between Dentin Samples and Sealers**

A Ramé-Hart goniometer (Ramé-hart Instrument Company, Netcong, NJ, USA) was used to measure the contact angle between treated dentin surfaces and the sealers AH Plus (Dentsply, Petrópolis, RJ, Brazil), Pulp Canal Sealer EWT (Kerr; Sybron Dental Specialties, Romulus, MI, USA) and MTA Fillapex (Angelus Indústria de Produtos Odontológicos S/A, Londrina, PR, Brazil).

The sealers were manipulated according to the manufacturer's specifications. One drop of sealer (0.1 mL) was deposited on each dentin surface with a 0.5 mL BD ultrafine syringe (Becton Dickinson, Franklin Lakes, NJ). For each group, 30 samples were evaluated (10 samples per sealer). The sealer wettability was monitored and computed during a period of 1 minute (60 measurements per minute).

The following formula was used to evaluate each sealer's dynamic wettability (SDW):[11]

$$SDW\ (\%) = \frac{(\text{initial angle} - \text{final angle})}{\text{initial angle}} \times 100$$

**Statistical Analysis**

Data were tabulated in Excel 2011 and were statistically analyzed using the SPSS program (IBM SPSS Statistics). The Kolmogorov-Smirnov normality test was applied to evaluate the distribution of the data. The data obtained were not normally distribution. Therefore, the nonparametric Kruskal-Wallis H and Mann-Whitney U tests ($p < 0.05$) were applied.

## Results

Table 1 shows the sealer dynamic wettability values (%) of the different experimental groups. In all sealers evaluated, the presence of a smear layer (control group) disfavored the dynamic wettability. Regarding AH Plus sealer, the use of 17% EDTA and QMix statistically improved the sealer wettability. Etidronate performed worse than EDTA and QMix, but better than control. Pulp Canal Sealer showed the same behavior. For MTA Fillapex, QMix presented the best results, followed by 17% EDTA. The result of 18% etidronate was statistically lower than the others and was similar to the control group.

**Table 1.** Sealer dynamic wettability values (%) after the different treatments

| | AH Plus (%) | Pulp Canal Sealer EWT (%) | MTA Fillapex (%) |
|---|---|---|---|
| **Control** | 3.4 ± 5.0[c] | 13.8 ± 2.8[c] | 16.8 ± 3.8[c] |
| **17%EDTA** | 8.4 ± 2.8[a] | 39.8 ± 2.0[a] | 22.5 ± 1.6[b] |
| **18%Etidronate** | 5.1 ± 1.2[b] | 33.4 ± 4.1[b] | 19.6 ± 2.2[c] |
| **QMix** | 10.8 ± 3.1[a] | 38.2 ± 5.5[a] | 28.8 ± 4.3[a] |

*Different letters[(a,b,c)] in the column indicate statistically significant values ($P<0.05$).

To demonstrate the action of each protocol used in the tested samples, we obtained photomicrographs, as shown in Figure 1, to evaluate the smear layer removal. The smear layer removal by EDTA and QMix was better than that obtained with 18% etidronate.

tions can change the dentin roughness. This roughness can contribute to wettability and adhesion. A surface can be expected do have greater wettability when it rougher due to the increased contact area, according to the Wenzel equation.[2]

The 17% EDTA was evaluated because it is a solution commonly used with positive results for smear layer removal.[6,12] We found that the use of EDTA favored the wettability of the sealers evaluated, possibly by increasing their roughness and surface energy.[15,16] Because EDTA has a deleterious erosive effect on dentin surface, etidronate and QMix are increasingly used for smear layer removal.[17]

Regarding the use of QMix, this combines the action of EDTA, opening the dentinal tubules, and the antimicrobial action of CHX in the same product, thus reducing the operator's working time while assuring ideal final irrigation.[18]

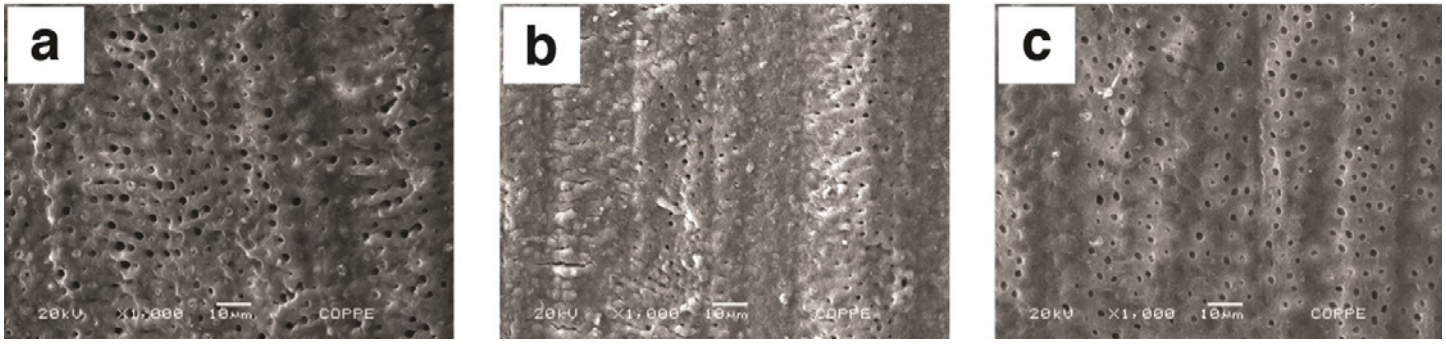


**Figure 1.** SEM 1000X. Surfaces irrigated with (a) 17% EDTA, (b) 18% etidronate and (c) QMix, as final active irrigant, showing the different quantities of open tubules

## Discussion

2% chlorhexidine has been leading choice as chemical auxiliary substance during preparation due to its antimicrobial effect, comparable to NaOCl,[12] associated with substantivity, which is the ability to bind to dental tissues and thus prolong the antimicrobial effect.[13] Various studies have evaluated the wettability of endodontic sealers after the use of NaOCl associated with chelating agents and acids. However, no study has evaluated the interaction between CHX and chelating agents and acids regarding sealer wettability. Thus, we evaluated the wettability of AH Plus, Pulp Canal Sealer EWT and MTA Fillapex sealers after the use of 2% chlorhexidine gel associated with EDTA, QMix or etidronate for smear layer removal.

All null hypotheses were rejected. The substances used after the 2% chlorhexidine gel influenced the sealer dynamic wettability. Wettability increased after the use of chelating substances, due to the removal of the smear layer, through exposure of dentinal tubules, increasing the area for penetration of chemicals and sealers.[5,14] These results are in agreement with Assis, Prado and Simão[15], who found that removing the smear layer led to reduction of the contact angle of the sealers AH Plus and Real Seal SE in contact with dentin previously treated with EDTA. Also, chelating solu-

The mixture of EDTA and CHX is known for producing a white precipitate, but in QMix, this does not happen due to its chemical structure.[19] Studies have shown that the QMix can be as effective as 6% sodium hypochlorite for bacterial clearance.[20,21] Comparing the effect of QMix to EDTA to remove the smear layer, according to the literature, QMix acts as effectively as 17% EDTA based on the number of open dentinal tubules.[7,20,22,23]

In the present study, the use of QMix showed similar results to EDTA for the sealers AH Plus and Pulp Canal EWT and was superior to MTA Fillapex. The reason for the satisfactory wettability with the use of QMix is associated to the action of EDTA to remove the smear layer and the presence of a surfactant agent combined with CHX.[20] The surfactant reduces surface tension, which is the result of intermolecular attraction of a liquid in contact with a solid surface. When this intermolecular attraction is weakened, the surface tension decreases by increasing its wettability.[24] Further, the use of CHX leads to an increase of the free energy of the dentin surface and dentin wettability, and a decrease of endodontic sealer contact angle.[25] The better results obtained when evaluating the wettability of the MTA Fillapex, after the use of QMix, can be associated with the fact that this is a hydrophilic sealer and chlorhexidine makes the surface even more hydrophilic.[26,27]

As was expected from the literature, the etidronate proved

to be a weak agent for calcium complexation, the reason it causes less change in the dentin than other chelating agents. HEBP as final irrigant seems to be a promising solution with less impact on the root dentin mineral content.[28] Besides that, studies have shown that this substance does not have negative interaction with other irrigants.[8,10,29] In this study, its use produced worse than other solutions. As observed in Figure 1, the lower wettability can be associated with the reduced action of this agent for removal of the smear layer, meaning less surface roughness.[9,28,29] Although there is generally lower wettability of sealers, de Deus *et al*.[10] showed that chelating agents like EDTA and citric acid produced an overly deep zone of demineralized dentin to allow efficient sealer penetration, making HEBP a promising chelating agent to increase the filling material's bonding strength to dentin.

The time of 3 minutes used in irrigation protocols of the chelating substances for removal of the smear layer was chosen in accordance with previous studies, which suggest this interval is best for more effective action of EDTA and QMix.[20,30] Because of the self-limiting effect of EDTA, in this study solutions were renewed every minute, thus achieving more effective action.

## Conclusion

In conclusion, the solutions used for smear layer removal, after the use of chlorhexidine during preparation, influenced the wettability of AH Plus, Pulp Canal Sealer EWT and MTA Fillapex sealers. Furthermore, the QMix 2in1 irrigating solution showed the best activity among the with other irrigants tested, which could be an advantage in terms of its use in regular clinical practice.

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## Mini Curriculum and Author's Contribution

1. Iris Sol Figueiredo Telles – DDS. Contribution: bibliographical research; experimental procedures; manuscript writing; critical review and final approval.
2. Marcello Ghetti de Melo – DDS. Contribution: effective scientific and intellectual participation in the study; manuscript writing; critical review and final approval.
3. Marcela Baraúna Magno – DDS and MSc. Contribution: experimental procedures; manuscript writing; critical review and final approval.
4. Renata Antoun Simão – PhD. Contribution: effective scientific and intellectual participation in the study; manuscript writing; critical review and final approval.
5. Maíra Prado – DDS and PhD. Contribution: effective scientific and intellectual participation in the study; manuscript writing; critical review and final approval.

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