



UNIVERSIDADE FEDERAL DO PARÁ  
INSTITUTO DE CIÊNCIAS DA SAÚDE  
PROGRAMA DE PÓS-GRADUAÇÃO EM ODONTOLOGIA - MESTRADO

**Etidronic acid, a weak chelator: Wettability of an epoxy resin-based root canal sealer on  
intraradicular dentin treated with different protocols**

Marcela Baraúna Magno

Belém –Pará

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Dissertação apresentada ao Curso de Odontologia da Universidade Federal do Pará, como pré-requisito para a obtenção do título de Mestre em Odontologia, pelo programa de Pós-Graduação em Clínica Odontológica.

Área de Concentração: Clínica Odontológica

Orientador: Prof. Dr. Oscar Faciola Pessoa

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“Se sua mente pode conceber e seu coração acreditar,  
...você pode alcançar.” (William Arthur Ward)

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**Etidronic acid, a weak chelator: Wettability of an epoxy resin-based root canal sealer on intraradicular dentin treated with different protocols**

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**Keywords:** Chelating agents, contact angle, EDTA, epoxy resin, etidronic acid, wettability

## **Etidronic acid, a weak chelator: Wettability of an epoxy resin-based root canal sealer on intraradicular dentin treated with different protocols**

**Purpose.** To investigate the wettability of AH Plus in contact with dentin treated with different weak chelating protocols involving etidronic acid (HEBP) and EDTA.

**Materials and Methods.** Fifty-six human root dentin slices were used. They were irrigated with 5.25% sodium hypochlorite (NaOCl) or a mixture of 5.25% NaOCl/18% HEBP and washed with distilled water (DW) to simulate irrigation during chemo-mechanical preparation. The samples irrigated with NaOCl were divided into 5 groups: G1-DW (control group); G2-17% EDTA; G3-17% EDTA + 2.5%NaOCl; G4-18% HEBP; and G5-18% HEBP + 2.5%NaOCl; the samples irrigated with the mixture NaOCl/HEBP were divided into 2 groups: G6-DW; G7-NaOCl/HEBP + 2.5%NaOCl. All of the protocols received irrigation with DW between the substances and as final rinse. The Rame-Hart goniometer was used to measure the contact angle between the dentin surfaces and AH Plus. The results were subjected to Kruskal-Wallis and Dunn tests ( $p < 0.05$ ).

**Results.** Compared with the control group, groups in which the smear layer was removed showed a lower contact angle ( $p < 0.05$ ), except for G7. The NaOCl/HEBP mixture (G6) showed the lowest contact angle of AH Plus, but the NaOCl final irrigation (G7) increased the angle. 17% EDTA (G2) and 18% HEBP (G4) have similar behaviour and final irrigation with NaOCl (G3 and G5) did not change wettability when these chelators were used.

**Conclusions.** 18% HEBP showed the potential to replace conventional treatment with 17% EDTA when mixed with NaOCl and used as the main irrigant, favouring the wettability of AH Plus.

**Keywords:** Chelating agents, contact angle, EDTA, epoxy resin, etidronic acid, wettability

### **Etidronic acid, a weak chelator: Wettability of an epoxy resin-based root canal sealer on intraradicular dentin treated with different protocols**

Endodontic treatment is a series of procedures that aims disinfection and sealing of the root canal system. The mechanical instrumentation produces a smear layer that adheres weakly to the root canal walls and obliterates dentinal tubules, reducing its permeability.<sup>12</sup> This layer has an adverse effect on dentin bonding, on the penetration of the irrigating solutions and the sealers to the dentinal tubules,<sup>25</sup> increasing microleakage after canal obturation.<sup>22</sup> Removing the smear layer from the root canal using irrigating solutions has been suggested to ensure that the root canal filling can adapt to the canal perfectly,<sup>12</sup> as well as to reduce the adherence of microorganisms to dentin, increasing the chance of success of endodontic therapy.<sup>22</sup>

The alternating use of sodium hypochlorite (NaOCl) and 17% EDTA is an efficient tool for removing the endodontic smear layer. NaOCl dissolves the necrotic tissue as well as the organic components of this layer, inactivates endotoxins, disintegrates endodontic biofilms and is the core chemical responsible for root canal debridement.<sup>33</sup> EDTA removes calcium ions ( $\text{Ca}^{2+}$ ) from the mineralised portion of the smear layer.<sup>10,26</sup> Ideally, the combination of agents would prevent formation of the smear layer on the dentin. However, hypochlorite is very reactive and cannot easily be combined with other chemicals, which makes this agent ineffective. The 1-hydroxyethylidene-1,1-bisphosphonate (HEBP), which is also known as etidronic acid, has been identified thus far as a decalcifying agent which compatible with NaOCl. This is a biocompatible chelator that can be mixed with NaOCl solution without any short-term loss in antimicrobial properties<sup>34</sup> and causes minimal changes in the ability of sodium hypochlorite to dissolve organic matter.<sup>29</sup> This mixture could be used as a single irrigant during and after instrumentation use so that a smear layer is created and immediately removed.<sup>10</sup>

The use of different agents cause alterations in the chemical and structural composition of human dentin.<sup>8,26,28</sup> EDTA is a well-known strong chelating agent, which can not only extract  $\text{Ca}^{2+}$  ions from the smear layer and dentin, but can also produce areas of demineralisation due to prolonged contact with surface dentin,<sup>11</sup> thus decreasing the calcium/phosphorus (Ca/P) ratio of root dentin.<sup>7</sup> These alterations may also negatively affect the adhesion and sealing ability of dental materials, such as root canal sealers, to dentin.<sup>25</sup> Therefore, a weak or moderate decalcifying agent may represent a good choice for preservation of the peritubular and

intertubular dentin. Lottanti et al.<sup>18</sup> suggested that etidronate has the potential to replace the conventional treatment with EDTA. Being a weak chelator, HEBP results in less damage to dentin.<sup>10</sup> Thus, it can condition radicular dentin to greater resin adhesion.<sup>9</sup>

In the context of adhesion, the materials must come into intimate contact with the substrate to allow either chemical adhesion or penetration for micromechanical surface attachment. Thus, wettability is one of the most important physicochemical properties that interfere with the ability of its penetration both into the main system of canals, and into the dentinal tubules. It is expressed by a contact angle between the drop of liquid and the plane surface of the solid, and shows the ability of the liquid to spread on this surface.<sup>16</sup> Root canal sealers with good flow ability and low surface tension spread and interact better with this surface, forming a low contact angle,<sup>17</sup> can be easily placed along the entire root canal and are capable of wetting the canal walls.<sup>27</sup>

Some studies have evaluated the wettability of endodontic sealers in dentin treated with chelators like EDTA<sup>2</sup> and maleic acid,<sup>4</sup> but there have been no available studies to date in the literature on root canal dentin treated with HEBP or weak chelators. Hence, the purpose of this study was to investigate the wettability of one epoxy resin-based sealer on root canal dentin treated with different irrigation protocols using HEBP, with and without final irrigant. A standard 17% EDTA solution was used as a reference for comparison. The null hypothesis was that the irrigation solutions do not influence sealer wettability, and the hypothesis tested was the use of a weak chelator increase AH Plus wettability.

## **MATERIALS AND METHODS**

This study was approved by the ethics Committee on Human Research

Twenty eight single-root human teeth, with minimum length of 11mm, were used. The sample size was determined after a pilot study where was attributed power test 80% and significance level 5%. Teeth with caries, cracks, and root dilacerations were excluded.

After extraction, the teeth were immediately placed in saline solution at 4°C after debridement of the surrounding soft tissue and debris. The crowns were removed at the cement-enamel junction and the remaining roots were split into buccal-lingual direction, separating each root into two halves, using a diamond disk (KG Sorensen Ind. e Com., Barueri,

SP, Brazil) at low speed under coolant water, which provided 56 samples. The two halves of the root were not used as matching samples. The surfaces were polished with a series of ascending grades (80, 100, 120, 150 and 180) of silicon carbide abrasive papers under water coolant to obtain standard surface roughness, and smooth and flat surface, to provide the analyses. Then, the samples were thoroughly washed and ultrasonicated in distilled water to remove residual particles.

The solution treatments were carried out in three stages. In stage 1, the specimens were irrigated, using a syringe, with 25mL of irrigating solutions (5.25% NaOCl or a mixture of 5.25% NaOCl/18% HEBP) to simulate the chemo mechanical preparation. In stage 2, the inorganic phase of the smear layer was removed through the immersion on chelating agents (17% EDTA for 3 minutes or 18% HEBP for 5 minutes). In step 3, surface final treatment was performed with the use of 2.5% NaOCl solution for 1 minute. Between each step and as final rinse, the specimens were washed with distilled water for 1 minute. The random scheme of protocols irrigations are shown in figure 1.

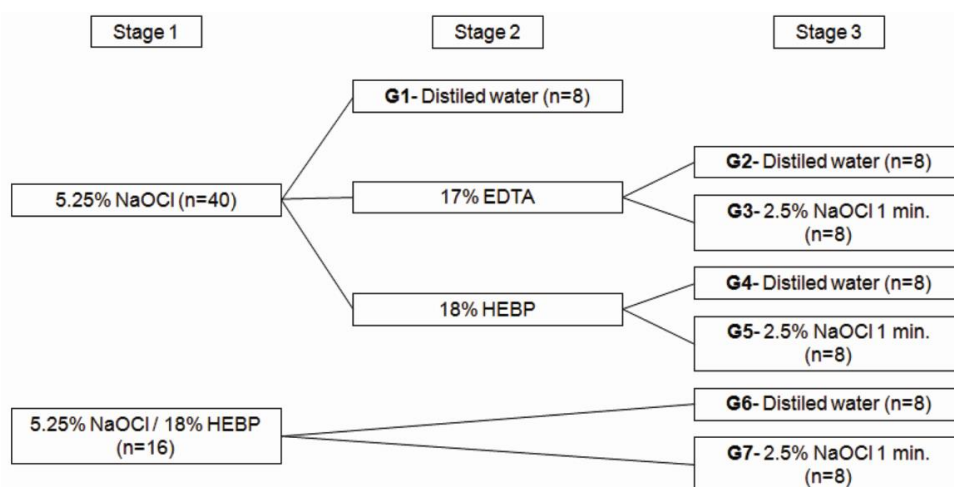


Figure 1. Irrigation regimes conducted for dentin samples

The 18% HEBP solution (Zschimmer & Schwarz Mohsdorf GmbH & Co KG, Burgstädt, SN, Germany) was prepared using the pure chemical dissolved in distilled water. The mixture 5.25% NaOCl + 18% HEBP was prepared using equal parts (1:1) of these two solutions associated with manual agitation, immediately before the experiments. All solutions were stored

at 5°C in dark containers after experiments. However, during the experiment, the solutions were kept at room temperature.

After treatment, the specimens were dried with paper points (Dentsply Ind & Com. LTDA, Petrópolis, RJ, Brazil) and the contact angle was measured.

### **Contact Angle measurement**

After completion of the treatments, the dentin specimens were positioned on microscopic lamel in a Rame-Hart goniometer (Rame-Hart Instrument Co, Netcong, NJ). This equipment has a flexible video system for measuring the static and dynamic contact angles and surface free energy with the sessile drop technique, and was used to measure the contact angle between AH Plus (Dentsply, Petropolis, RJ, Brazil) and treated dentin. The sealer was mixed according to the manufacturer's instructions. The goniometer was aligned and focused on the dentin-sealer interface.

At this point, a controlled-volume droplet (0.1 mL) of sealer was placed over the internal side of the root canal surface (intraradicular dentin) of a specimen from each group. The volume of the sealer was controlled by means of BD ultra-fine syringe of 0.5 mL/cc (Becton Dickinson, Franklin Lakes, NJ, EUA). Two drops were placed on each specimen. The spreading process was recorded and three images/second of each drop were analysed to provide the values of contact angles with the help of the Rame-Hart goniometer software (Rame-Hart Instrument Co, Netcong, NJ). Images of the final three seconds of each drop were analysed to provide the values of contact angles. All measurements were carried out by one calibrated operator. The data were computed with BioEstat 5.0.

All experiments were performed under standard environmental conditions.

### **Statistical Analysis**

Shapiro-Wilk test was used to check normality distribution. The sample exhibited abnormal distribution. Data were analysed with Kruskal-Wallis analysis of variance and Dunn tests ( $p < 0.05$ ) to compare the contact angle between groups.

## RESULTS

Table 1 lists the median (Med) and interquartile range (IQR) values of the contact angles between the sealer and treated dentin surfaces. By evaluating the contact angle between treated dentin surfaces and AH Plus, better spreading was observed for G6. The comparison of measured contact angle values before and after the application of chelating agents shows that when the smear layer was removed, the values of contact angle were lower, except for G7. The comparison between the use of chelating agents with and without NaOCl final irrigation showed that the final irrigation did not significantly change the contact angle, except for when comparing G6 and G7, where this final irrigation increased the contact angle. Representative static contact angles obtained for AH Plus on root canal dentine treated with different irrigant protocols are shown in Fig. 02.

Table 1. Median  $\pm$  interquartile range values of contact angle ( $A^\circ$ ) between treated dentin surfaces and AH Plus.

Groups	$A^\circ$
	Med $\pm$ IQR
G1 NaOCl	55.45 $\pm$ 3.825 <sup>A</sup>
G2 EDTA 17%	44.55 $\pm$ 1.425 <sup>B a 1</sup>
G3 EDTA 17% + NaOCl	42.7 $\pm$ 1.875 <sup>B 1</sup>
G4 HEBP 18%	46.55 $\pm$ 5.125 <sup>B a 2</sup>
G5 HEBP 18% +NaOCl	44.65 $\pm$ 4.225 <sup>B 2</sup>
G6 HEBP/NaOCl	31.75 $\pm$ 2.55 <sup>B b 3</sup>
G7 HEBP/NaOCl + NaOCl	50.25 $\pm$ 3.125 <sup>A 3*</sup>

Superscript uppercase letters (<sup>A,B</sup>) indicate statistically significant values between groups with (G1) and without smear layer. Superscript lowercase letters (<sup>a,b</sup>) indicate statistically significant values between the chelating agents (G2, G4, G6). The presence of \* next to a number indicates statistically significant values after final treatment between each chelating solution (G2/G3-1, G4/G5-2, G6/G7-3).

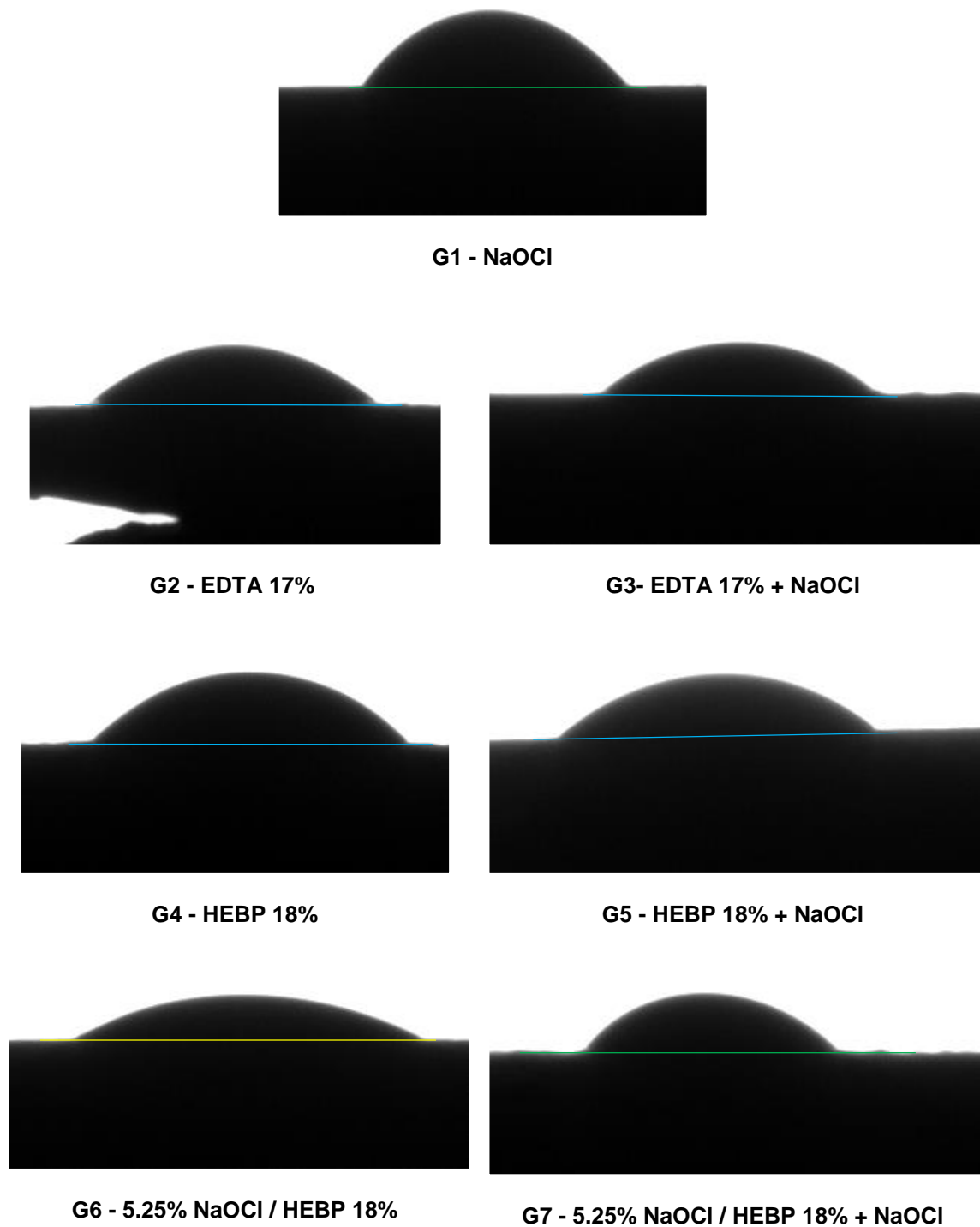


Figure 02. Representative images of sessile drops of AH Plus applied to root canal dentine treated with different irrigating solutions. Different colors indicate statistically significant values between groups.

## DISCUSSION

The physicochemical properties of a root canal sealer might characterize its clinical behavior during and after obturation of the root canal system. One of these properties is the optimal wetting, expressed with the formation of a contact angle. The contact angle has an inverse relationship with wettability; that is, the lower value of the angle, the greater is the wettability of the liquid and hence improved adhesion.<sup>4</sup> In the present study, bonding to root canal dentine was affected differently by the endodontic irrigant regimens. In general, improvements in wettability values were found in the following order: G6 > G3 > G2 > G5 > G4 > G7 > G1 as shown in Table 1.

The experiment was accomplished under standard environmental conditions and with a controlled volume of each drop sealer. This was done because the pH and temperature variations caused alterations in the surface tension levels of solutions,<sup>31</sup> and changes in the drop size could affect the value of the contact angle.<sup>13</sup>

Epoxy resin-based sealer cements, such as AH Plus, were used because of their acceptable physical properties and adequate biological performance.<sup>35</sup> Only two drops were placed because the root canal surface has a very limited area on which to place more droplets.

For correct measurement of the contact angle, the surface must be clean, flat and smooth,<sup>19</sup> although the root canal dentin surfaces were not smooth after cleaning and shaping. Therefore, for this study, the fine polishing was a substitute for graver paper, to obtain surfaces that were somewhat similar to clinical conditions before instrumentation, as reported in other studies.<sup>2,32</sup>

Comparing groups with (G1) and without (G2, G3, G4, G5, G6) a smear layer, groups in which the smear layer was removed showed a lower contact angle, except for G7, that presents similarity statistic to G1. This has also been shown in other studies<sup>2,32</sup> and, knowing that roughness has a significant contribution to the wetting behaviour of a surface,<sup>15,24</sup> this can be explained by the action of chelating solutions with regard to removing the smear layer and exposing the dentinal tubules,<sup>18</sup> thus increasing the roughness of the dentin surface.<sup>28</sup> According to the Wenzel equation, this action increases the wettability of the sealer.<sup>30</sup> The increase in surface roughness favours the micromechanical retention of materials; moreover, this facilitates bacterial adhesion.<sup>23</sup>

Dentin is composed of two different substrates: hydroxyapatite, which has a high surface energy, and collagen, which has a low surface energy.<sup>1</sup> AH Plus is able to bond to the organic phase of root dentin, most likely in the collagen fibres.<sup>20</sup> Knowing that EDTA is a more powerful agent for removing the smear layer and hydroxyapatite than HEBP,<sup>10,34</sup> this is able to remove more  $\text{Ca}^{2+}$  ions than HEBP (G4),<sup>7</sup> exposing more of the collagen network, which is better for the bonding sealer. Although EDTA and HEBP result in structurally different surfaces, this does not significantly influence AH Plus wettability. The time of action of HEBP (5 minutes) compared to EDTA (3 minutes) may have influenced on final wettability. These different time of action for solutions was determined based on smear layer remove and open dentin tubules.<sup>10</sup>

The best spreading of the sealer in G6 is related to the higher dentin roughness produced for this irrigation protocol, when compared to other substances, as shown by Tartari et al.<sup>28</sup> Another study found that the use of a mixture of NaOCl/HEBP during irrigation had a good significant impact on the bond strength of the epoxy-based sealer to root dentin.<sup>21</sup> This can be explained by three factors: (i) the ability of NaOCl deproteinate and create channels in dentin,<sup>14</sup> increasing the contact area for the action of HEBP and the roughness dentin; (ii) the weak chelating but continued action of HEBP probably removed a greater amount of  $\text{Ca}^{+}$  (compared to G2 and G4),<sup>7</sup> exposing more collagen fibres, making the surface favourable for this sealer; and (iii) the fact that each sample of this group was irrigated with 25ml of the solution mixture and, in the other groups, the samples were immersed in 40ml of chelator, which may have increased the time of action and the substance effect.<sup>6</sup>

After debridement and disinfection, negative bacterial cultures only occur in 40–60% of cases; microorganisms can partially survive chemo-mechanical preparation inside dentinal tubules and irregularities of the root canal. Therefore, after removal of the smear layer, it is rational to complete irrigation with another disinfectant to attack the remaining bacteria. A low concentration of NaOCl is used with this aim.<sup>5</sup> The final irrigation with 2.5% NaOCl, after the addition of 17% EDTA (G3) and 18% HEBP (G5), did not cause any changes in the wettability of the sealer, which is in disagreement with the results of Assis et al.,<sup>2</sup> that find significant alterations in EDTA group. This contradiction can probably be explained because the samples were dried with nitrogen gas by Assis,<sup>2</sup> while in the present study they were dried with paper points to simulate clinical environments. This might have caused alterations regarding surface

free energy, because contact angle measurements are highly sensitive. In HEBP group, the probably explanation is that HEBP was not able to expose a significant amount of the collagen network for NaOCl deproteinisation action. However, the wettability was decreased with final 2.5% NaOCl irrigation after HEBP/NaOCl treatment (G7). This probably occurred because the organic-dissolving properties of NaOCl on the collagen components of dentin after removal of the smear layer<sup>14</sup> resulted in dentin surfaces similar to etched enamel and led to a high energy surface,<sup>3</sup> which was unfavourable for AH Plus.

Knowing that different irrigant solutions affect directly dentin morphology and wettability to different sealers; and the first step of the interaction between adhesive and substrate is the wetting of the dentin,<sup>4</sup> this effect should be taken into account. The present study highlights the good effect of etidronic acid on the wettability of epoxi-resin based sealer on to the root canal dentine, specially when used as main irrigant, which is required for obtaining good adhesion and obturation seal.

## **CONCLUSIONS**

Under the experimental conditions, the present study highlights the good effect of the mixture NaOCl/HEBP on the wettability of epoxi-resin based sealer on to the root canal dentine, specially when used as main irrigant, which is required for obtaining good adhesion and obturation seal.

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## **CLINICAL RELEVANCE**

The improvement in wettability and adhesion of AH Plus when a mixture 5.25% NaOCl / 18% HEBP is used is specially relevant in single cone obturation techniques, in which the sealer must play a more incisive role on the filling of spaces between the cone and dentinal walls.

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