

# Wettability of Irrigants used in Root Canal Treatment

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## Abstract

This study assessed the contact angle and the surface tension of different chemical solutions used in the chemomechanical preparation of root canals in order to calculate its wettability. The tested irrigants were: 2.5% sodium hypochlorite (NaOCl); 2% chlorhexidine (CHX); BioPure MTAD<sup>®</sup>; and 0.9% sterile physiological saline solution, as the control. A goniometer was used to measure the contact angle and the surface tension. Then the wettability of each solution was calculated using Young's equation. BioPure MTAD<sup>®</sup> showed significantly higher contact angle ( $p=0.002$ ), compared to the other substances, which did not differ among themselves. As the surface tension, BioPure MTAD<sup>®</sup> showed significantly lower values ( $p=0.001$ ). As for 2.5% sodium hypochlorite, 2% chlorhexidine, and the control, chlorhexidine presented lowest surface tension ( $p<0.01$ ). BioPure MTAD<sup>®</sup> showed the best wettability in relation to the other tested irrigant, followed by two percent of CHX and 2.5% NaOCl.

**Keywords:** Contact angle; irrigants; surface tension; wettability; Young's equation

## Introduction

The main objective of dental root canal treatment is to achieve a canal system free of microorganisms. The irrigant solutions used during treatment have the purpose of eliminating bacteria from infected root canals and promoting dissolution of the pulp tissue (a dense connective tissue). For decades, researchers have searched for antimicrobial agents that can effectively debride the root canal system. Until now the ideal chemical substance remains unknown [1-3].

Among the solutions available for root canal irrigation, two are the most commonly employed and one was more recent introduced as a promising irrigant. The first is the sodium hypochlorite (NaOCl), a halogen substance, which has pronounced antimicrobial activity and tissue solvent capacity. Second is the chlorhexidine (CHX), which is a cationic bisbiguanida with antibacterial property, derived from its electrostatic attraction by the microbial cell wall, which damages the cell osmotic balance. CHX also has substantivity due to its capacity to bind in negatively charged surfaces on the oral cavity, such as the mineralizing dental tissue, acquired pellicle, mucous membranes and restorative materials, and be slowly released. Finally, the new intracanal irrigant is the Biopure MTAD<sup>®</sup>. This product is an aqueous solution of doxycycline, a broad-spectrum antibiotic; citric acid, a demineralizing agent; and polysorbate 80 detergent [4].

An irrigant solution used in root canal preparation must be in contact with the canal wall to promote the dissolution of organic tissues and disinfect the dentin and its tubules. During root canal instrumentation, untouched areas are a common problem even with superelastic rotary preparation. For example, studies with mesiobuccal root canals of maxillary first molars reported an average of untouched areas of 40.0%-47.4% [5-7]. For this reason, the solvent action and the antimicrobial activity in these areas are crucial to root canal disinfection.

A chemical solution for endodontic use must exhibit substantial wetness, which increases its solvent capability and improves antimicrobial activity in non-instrumented areas of the root canal system [8]. The wettability of a liquid can be assessed by measuring the contact angle of the liquid with a solid surface and its surface (or interfacial) tension. The present study was conducted to assess the contact angle and the surface tension of NaOCl, CHX and Biopure MTAD<sup>®</sup> in order to calculate there wettability.

## Material and Methods

For this experiment, the following solutions were tested: 2.5% NaOCl (Q'boa<sup>®</sup>, Indústrias Anhembi S/A, Osaco, SP, Brazil) 2% CHX (Fórmula Base Farmácia de Manipulação Ltda, Rio de Janeiro, RJ, Brazil), Biopure MTAD<sup>®</sup> (3% doxycycline, 4.25% citric acid, and 0.5% polysorbate 80) (Dentsply, Tulsa, OK, USA), and 0.9% sterile physiological saline solution (control) (Equiplex, Aparecida de Goiânia, GO, Brazil).

## Measurement of contact angle

The contact angle was measured on glass slides with a 1- $\mu$ l drop of the each chemical solution using a goniometer (First Ten Angstroms, Atlanta, GA, USA), which belongs to the Biomaterials Laboratory of the Military Institute of Engineering (IME, Rio de Janeiro, Brazil). For each chemical solution, 15 microscope glass slides with a size of 20  $\times$  20 mm and thickness of 0.13–0.17 mm (Lamedid Comercial e Serviços LTDA, Barueri, SP, Brazil) were used.

After being formed, the droplet was placed on the slide as soon as it came in contact with the surface and detached from the syringe. Each droplet was photographed after being placed on the slide, and the image was digitized with a scanner. Then, the contact angle between the droplet and the slide was calculated by the software Fta32\_Video (First Ten Angstroms, Atlanta, GA, USA) using the tangent of the angle between the droplet and the solid surface.

## Measurement of surface tension

The surface tension of each solution was measured with the pending droplet method, using the same software described above. In this method, the liquid droplet stays attached to the goniometer

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syringe, and the surface tension is determined by fitting the shape of the drop (in a captured video image) to the Young-Laplace equation, which relates surface tension to drop shape. The software does this automatically. In Young-Laplace equation  $\gamma (1/R_1 + 1/R_2) = \Delta P$ ,  $\gamma$  is the surface tension,  $R_1$  and  $R_2$  are the two principal radii of curvature of the surface, and  $\Delta P$  is the pressure difference across the interface.

In order to measure the surface tension, 10 droplets of each substance were used, which were then put in a glass receptacle and discarded. The droplets were formed by 1  $\mu$ l of the solution. Next, they were photographed, the images were digitized with a scanner, and the software calculated the surface tension. The results are expressed in millinewtons per meter (mN/m).

### Calculation of wettability

In order to assess the wettability of the solutions, Young's equation was used:  $\gamma_{LV} \cos\theta = \gamma_{SV} - \gamma_{SL}$ , where  $\gamma_{SL}$  represents the surface tension between the solid and the liquid,  $\gamma_{SV}$  and  $\gamma_{LV}$  are the surface tension of the solid and the surface tension of the liquid in equilibrium with the air, respectively, and  $\theta$  is the contact angle (Figure 1). For this purpose, the contact angle means computed for each solution and the surface tension means were used. The surface tension value of the solid in relation to the air used in this study was 1.05 J/m<sup>2</sup> in accordance with a previous study [9]. The surface tension of the solid in relation to the air ( $\gamma_{SV}$ ) was converted into millinewtons per meter in order to apply the equation.

The parametric one-way analysis of variance (ANOVA) was used to identify significant differences among the irrigant solutions. Tukey's post hoc was used to determine which irrigant were significantly different. The significance level was set at  $p < 0.05$ .

### Results

Table 1 shows the mean values of contact angle registered for the different irrigant solutions. The variance analysis (ANOVA) revealed that there is a statistically significant difference among the tested irrigants ( $p = 0.002$ ). Thus, the Tukey test showed that 2% CLX, 2.5% NaOCl, and the control substance were not different from each other but that they had a significantly lower contact angle than Biopure MTAD®.

Table 2 shows the mean values of surface tension registered for the different irrigant solutions. There is a statistically significant difference among the tested irrigants ( $p = 0.001$ ). The Tukey test showed that Biopure MTAD® presented a surface tension significantly lower than that of 2% CLX, 2.5% NaOCl, and the control substance. As for 2% CLX, 2.5% NaOCl and the control, CLX had the lowest surface tension ( $p < 0.01$ ).

The wettability of the substances, which were analyzed using Young's equation, is expressed in Table 3. Biopure MTAD® presented the highest wettability, followed by 2% CHX, 2.5% NaOCl, and the control solution.

### Discussion

The contact angle is an indicator of the wettability of irrigants. The experimental values of the contact angle can be obtained by direct measurement of the tangent of the angle between the liquid droplet and the solid surface of the substrate [10].

A low value of the contact angle indicates that the liquid has a high wettability, whereas a high value indicates a low wettability. It has been determined that if the contact angle is less than 90°, there is wetting of

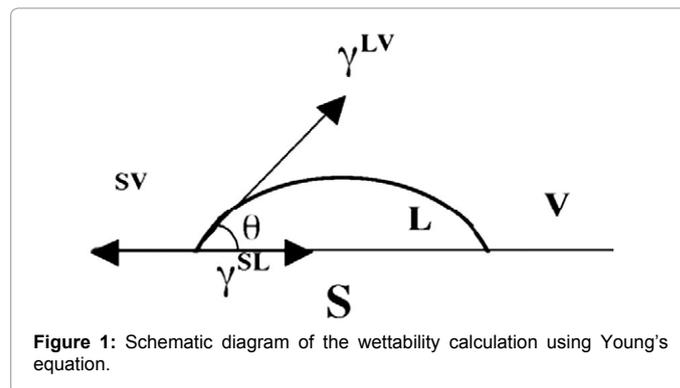


Figure 1: Schematic diagram of the wettability calculation using Young's equation.

Solution	Contact Angle
Chlorhexidine 2%	20.83° (7.23°) <sup>a</sup>
NaOCl 2.5%	21.25° (6.04°) <sup>a</sup>
Biopure MTAD®	28.34° (5.07°) <sup>b</sup>
Control	17.50° (4.46°) <sup>a</sup>
Different letters indicate statistically different values ( $p < 0.05$ )	

Table 1: Average contact angle values of the assayed substances.

Solution	Surface Tension (mN/m)
Chlorhexidine 2%	146.61 (12.67) <sup>b</sup>
NaOCl 2.5%	177.39 (14.49) <sup>a</sup>
Biopure MTAD®	90.55 (2.82) <sup>c</sup>
Control	186.87 (13.16) <sup>a</sup>
Different letters indicate statistically different values ( $p < 0.05$ )	

Table 2: Average surface tension values (mN/m) of the assayed substances.

Solutions	NaOCl at 2.5%	CLX2%	Biopure MTAD®	Control
Results	885.03	913.66	970.32	872.48

Table 3: Wettability values (mN/m).

the surface; if it is greater than 90°, there is no wetting; and a contact angle equal to zero represents complete wetting [11]. In the present study all tested substances presented contact angles at least three times lower than 90°.

The present study evaluated the contact angle and surface tension of the tested irrigants on glass slides and then the wettability was calculated using a mathematics equation. Some studies evaluated the effect of pre-treatment of dentin samples with different irrigants on the contact angle of the substance with the dentin, in other words, the wettability of the surface was the one assessed [12-14]. In one of these studies, the values for contact angles were significantly reduced after the pre-treatment of the dentin for 10 min with 5.25% NaOCl and 3% H<sub>2</sub>O<sub>2</sub> in comparison with 17% EDTA. The latter substance showed no significant difference compared to the control (distilled water) [12]. Another study using similar methodology did not find difference in wettability after pre-treatment of dentin with 2.5% NaOCl or 17% EDTA [14]. Despite the relevance of testing the wettability of dentin treated with different substances, the difficulty of standardizing this surface/substrate may explain the discrepancy among the results from different studies. In the present study, the standardization of the surface allowed the comparison of the wettability of solutions controlling this bias.

In this work, surface tension was measured using the pending droplet method. Recently, this approach was used to assess the effect of the addition of benzalkonium chloride to sodium hypochlorite on

its wetting properties, contact angle, and surface energy [15]. For the Biopure MTAD® versus NaOCl, the present study confirmed a previous report that the surface tension of Biopure MTAD® is lower than the NaOCl, despite the fact that the tested concentration of NaOCl used in this study was half of the used in an early study (5,25%) [8], In relation to CHX, there has been no previous report about surface tension of this irrigant at a concentration of 2%. In the present study, CHX assumed the intermediate position regarding the surface tension.

Surface tension is considered to be one of the most important factors in determining the wettability of a solution. Consequently, it is expected that the solution with the lowest surface tension can wet the walls of the root canal more extensively than those with a higher value can [16]. This hypothesis was confirmed in the present study.

Wettability is the tendency of a given fluid to spread over or adhere to a solid surface. The wetting is correlated to the contact angle  $\theta$ , while the spreading is related to the extension of the projected area [1]. This property is important for the chemical solution to penetrate the main and lateral canals, as well as the dentinal tubules, and it is dependent upon the surface tension [8,17]. In the present study, Biopure MTAD® was the substance with the highest wettability followed by 2.5% NaOCl and 2% CHX. Another study pointed a result of surface tension favorable to Biopure MTAD® compared to NaOCl [8].

In the present work, the selected irrigants were tested pure, without the addition of any substance. Surface-active agents can reduce the surface tension of some irrigants up to 50%, increasing their wettability on a surface. This is a good strategy to increase the ability of the irrigant to penetrate the root canal dentin and, consequently, increase its antimicrobial action. Temperature and pH also affect the surface tension of solutions. Increasing the temperature or reducing the pH decreases the surface tension level [14,18,19].

Biopure MTAD® was launched in the market with the purpose of combining a higher detergent effect with strong antimicrobial efficacy. Although the wettability of Biopure MTAD® is superior than the other solutions tested, studies show that its antimicrobial and antifungal activity is lower than the 5% or 6% NaOCl and 2% CHX [20,21]. However, one study showed that MTAD was significantly more effective in killing *E. faecalis* than NaOCl when the solutions were diluted [22]. For these reasons some authors proposed the association of the antimicrobial peptide nisin in the composition of Biopure MTAD®. They found a significantly improvement on the bactericidal activity of Biopure MTAD® against *E. faecalis* [23]. Currently, the use of Biopure MTAD® as intracanal irrigant is more restrict to final irrigation, after the use of NaOCl as main irrigant [24]. There is no doubt that the ideal irrigant solution must present good wettability associated to effective antimicrobial activity.

## Conclusion

BioPure MTAD® showed the best wettability in relation to the other tested irrigant, followed by two percent of CHX and 2.5% NaOCl.

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