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ARTICOLO ORIGINALE

Comparative wettability of different sodium hypochlorite solutions

Analisi comparativa della bagnabilità di diverse soluzioni di ipoclorito di sodio

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Received 6 December 2011; accepted 20 May 2012

Available online 29 June 2012

KEY WORDS

Surfactants;
Wettability;
Sodium hypochlorite;
Contact angle;
Surface tension.

PAROLE CHIAVE

Tensioattivi;
Bagnabilità;
Ipoclorito di sodio;
Angolo di contatto;
Tensione superficiale.

Summary

Objectives: The present study evaluated the dentin wettability of sodium hypochlorite (NaOCl) with and without the addition of surfactants.

Materials and methods: Twelve premolars roots were longitudinally split using a low-speed rotary diamond saw. The 24 specimens were randomly distributed into four experimental groups: Group 1 = 5.25% NaOCl; Group 2 = Hypoclean; Group 3 = Chlor-Xtra; Group 4 = freshly produced MilliQ water (distilled water). Three different contact angles were measured: the sessile angle, the advancing angle, and the receding angle. All angles were measured with a goniometer Krüss G 23.

Results: The highest contact angle was observed in Group 4 (control group), considering both sessile and advancing angles. Hypoclean and Chlor-Xtra solutions immediately spread on the dentin surface, yielding a zero-degree contact angle. Pure NaOCl solution showed a 35-degree sessile contact angle and a 64-degree advancing contact angle. The detected receding angle was zero.

Conclusions: Obtained data clearly showed the superior wettability of Hypoclean and Chlor-Xtra in comparison to pure NaOCl and distilled water.

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Riassunto

Obiettivi: Il presente studio ha valutato il comportamento di diverse soluzioni di ipoclorito di sodio (NaOCl), con e senza l'aggiunta di tensioattivi, a contatto con la dentina.

Materiali e metodi: Dodici premolari monocanalari sono stati separati secondo l'asse longitudinale con un disco diamantato in rotazione a bassa velocità. I 24 campioni ottenuti sono stati distribuiti in modo casuale in quattro gruppi sperimentali: Gruppo 1 = NaOCl al 5,25%; Gruppo 2 = NaOCl al 5,25% con tensioattivi (Hypoclean); Gruppo 3 = NaOCl <6% con tensioattivi (Chlor-Xtra); Gruppo di controllo = acqua distillata (MilliQ 18 MOhm). Al fine di valutare la bagnabilità delle soluzioni nei confronti della dentina sono state effettuate tre misurazioni: l'angolo di contatto sessile, l'angolo di contatto di avanzamento e l'angolo di contatto di recessione. Tutti gli angoli sono stati misurati con un goniometro Krüss G 23.

Risultati: L'angolo di contatto più alto è stato osservato nel gruppo di controllo, considerando i valori sia dell'angolo di contatto sessile sia dell'angolo di contatto di avanzamento. La diffusione sulla superficie dentinale delle soluzioni del Gruppo 2 (Hypoclean) e del Gruppo 3 (Chlor-Xtra) è stata immediata, in accordo al valore registrato di zero gradi per i tre differenti angoli di contatto. Il Gruppo 1 (ipoclorito di sodio al 5,25%) ha mostrato un angolo di contatto sessile pari a 35 gradi e un angolo di contatto di avanzamento pari a 64 gradi; l'angolo di contatto di recessione rilevato è risultato pari a zero.

Conclusioni: I dati ottenuti hanno dimostrato chiaramente la superiore bagnabilità delle soluzioni di ipoclorito di sodio addizionate con tensioattivi nei confronti della dentina rispetto a quella ottenibile con solo ipoclorito di sodio al 5,25% o con acqua distillata.

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Introduction

Success or prognosis of root canal therapy has been shown to depend on many factors, including the quality of instrumentation and irrigation which interfere in the achieved disinfection of the root canal. Several studies reported poor debridement of the root canal regardless of the instrumentation technique used [1–3]. Using the reliable micro-tomography method, high percentage of untouched root dentin surfaces after root canal instrumentation has been shown [1,3]. Deficient mechanical preparation of the current chemical-mechanical methods could offer an opportunity for microorganisms to recolonize the filled canal space, resulting in endodontic failure. Both instruments and irrigants are used in conjunction to achieve endodontic debridement, mainly in cases of teeth presenting complex anatomy, in which the anatomic irregularities are commonly untouched by instrumentation [4].

Among the solutions used in root canal therapy, sodium hypochlorite (NaOCl) appears to fulfill most of the requirements for an ideal root canal irrigant [5]. It presents high organic tissue-dissolving ability [6–8]. Sodium hypochlorite also presents a wide-spectrum antimicrobial activity [6,9,10], killing endodontic pathogens even when organized in biofilms [11]. However, despite its excellent tissue-dissolving and antimicrobial abilities, NaOCl presents a relative high surface tension (48.90 mJ/m^2), limiting the solution penetration into canal irregularities and deeply into the dentinal tubules [12,13].

An ideal root canal irrigant should present the best contact with the dentin walls and debris. This contact depends on the dentin wetting ability of solutions [14]. In general, the wettability of a solution depends on the relationship between its surface tension and the surface tension of the surface to be wet [15]. Surface tension, or the excess force between molecules at interfaces, produces a tendency for a liquid surface area to decrease [16].

In wetting phenomena, this force controls the spreading of a liquid over a surface. The effects of the combination of surface energy and surface microtopography on wetting is a classical problem of physical chemistry of surfaces, discussed since the correction by Wenzel of Young's equation in 1932 [15]. Chemical and topographical heterogeneity of the substrate surface results in contact angle hysteresis [17,18]. That is, on heterogeneous surfaces such as dentin, rather than a single contact angle as anticipated from Young's equation of ideal surfaces, a continuum range of angles can be detected [19,20]. This requires special care in wetting measurements, and it is often forgotten in the relevant literature.

The effectiveness of an endodontic irrigant could be improved by reducing its surface tension because its wettability can interfere with the solution penetration in the complex anatomy of root canal system and also in dentinal tubules [14–16]. The ability of surface-active agents (surfactants) to reduce the surface tension of NaOCl solutions has been recently demonstrated [21,22]. Hypoclean (Ogna Lab Srl, Muggiò, Milan, Italy) is a commercial available NaOCl solution added with two surface-active agents, which presents low surface tension (29.13 mJ/m^2). In a recent study, Hypoclean showed an optimized ability to kill bacteria compared to a pure 5.25% NaOCl solution [23]. Cameron [16] demonstrated that the addition of surface modifiers enhanced the ability of NaOCl to dissolve organic material. Since the contact angle of a solution with dentin is directly related to its wetting properties, the present study evaluated the dentin wettability of NaOCl with and without the addition of surfactants.

Materials and methods

Three endodontic irrigant solutions were tested: 5.25% NaOCl (Nicolor 5, Ogna Lab Srl, Muggiò, Milan, Italy), <6% NaOCl

containing surface-active agents (Chlor-Xtra, Vista Dental Products, Racine, WI, USA), and 5.25% NaOCl containing surface-active agents (Hypoclean, Oigna Lab Srl, Muggiò, Milan, Italy). Freshly obtained MilliQ water (MilliQ 18 MOhm, Millipore Corporation, Billerica, MA, USA) was used in the control group.

Twelve extracted human premolars were used in the study. Teeth had been extracted for orthodontic purposes and did not present caries or restorations. After cutting off the crown and the apical third of each tooth, the remaining root was longitudinally split using a low-speed rotary diamond saw. Twenty four sectioned root halves were obtained.

Each cut surface was polished using a series of abrasive papers (CarbiMet; Buehler, Lake Bluff, IL) in the following sequence: 120/P120, 180/P180, 240/P280, 320/P400, 400/P800, and 600/P1200.

The 24 specimens were randomly distributed into three experimental groups and one control group: Group 1 = 5.25% NaOCl; Group 2 = Hypoclean; Group 3 = Chlor-Xtra; Group 4 = freshly produced MilliQ water (MilliQ, 18 MOhm). Contact angle measurements were carried out with a goniometer Krüss G 23 (Krüss GmbH, Hamburg, Germany).

Because of the non-ideal nature of the dentin surface, three different angles were measured as previously described [19]: the sessile angle (θ), obtained by gently placing about 1 mm-diameter droplets on the sample surface; the advancing angle (θ_a), obtained by increasing the drop volume until promoting the movement of the three-phase boundary; the receding angle (θ_r) obtained by decreasing the drop volume until promoting the retraction of the three-phase boundary (fig. 1). All angles were measured with a goniometer Krüss G 23.

One mm-diameter droplets of hypochlorite solutions or distilled water (control) were placed on coronal root dentin

using a new micro-syringe for each tested irrigant, and the sessile, advancing and receding angles were immediately measured by increasing or decreasing the drop volume, while keeping the syringe needle in the drop, as widely described in the chemico-physical literature on this subject [19,20].

Results

Contact angle measurements showed marked differences between the evaluated solutions (table 1). The highest contact angle was observed in Group 4 (control group), considering both sessile and advancing angles. The detected receding angle was zero.

Hypoclean and Chlor-Xtra solutions immediately spread on the dentin surface, yielding a zero-degree contact angle. Capillary penetration, resulting from the combination of surface topography and interface energy, drives the wetting liquids into the pores and crevices of the dentin substrate. In this case, it was not possible to measure advancing and receding angles, since the wetting pattern was completely controlled by capillary penetration. Pure NaOCl solution showed a 35-degree sessile contact angle, and a 64-degree advancing contact angle. The detected receding angle was zero.

Discussion

Shaping and cleaning root canals is challenging, considering that much of the root canal space is not totally accessible to instruments and irrigation solutions [1–4]. Consequently, the quality of root debridement can be influenced by irrigation solutions associated with root canal instrumentation. Sodium hypochlorite is widely used to disinfect root canals during

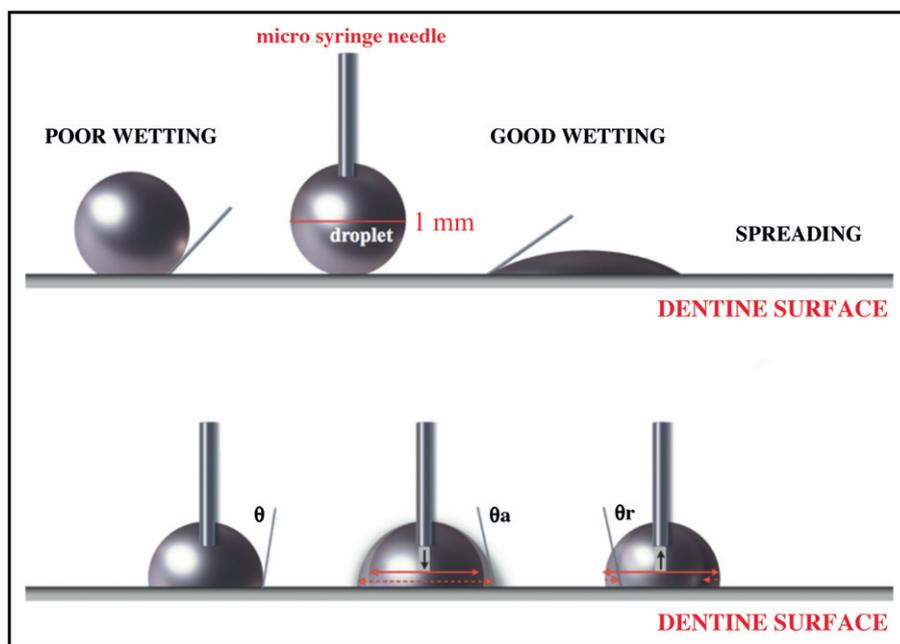


Figure 1 Schematic view of wetting and contact angles on dentin. θ , sessile contact angle; θ_a , advancing contact angle; θ_r , receding contact angle.

Table 1 Values obtained for sessile (θ), advancing (θ_a) and receding (θ_r) contact angles with the tested solutions.

Groups	Contact angles		
	θ	θ_a	θ_r
1	35 ± 3	64 ± 3	0
2	S	S	S
3	S	S	S
4	60 ± 10	85 ± 7	0

Group 1 = 5.25% NaOCl; Group 2 = Hypoclean; Group 3 = Chlor-Xtra; Group 4 = MilliQ water. S, spreading.

endodontic therapy, and it is usually employed at concentrations ranging from 0.5% to 5.25%. Nevertheless, despite the excellent tissue-dissolving [6–8] and antimicrobial properties [6,9,10], it presents a relative high surface tension (48.90 mJ/m²) [13]. The ability of surface-active agents (surfactants) to reduce the surface tension of NaOCl solutions has been recently demonstrated [16,21,22]. The tested liquids in Group 2 (Hypoclean) and Group 3 (Chlor-Xtra) are commercially available NaOCl solutions with a surface-active agent added, which present low surface tension (29,13 mJ/m² and 33,14 mJ/m², respectively) [24]. The tested solution in Group 2 had already showed increased ability to kill bacteria compared to pure 5.25% NaOCl solution [23]. The antimicrobial effect of Chlor-Xtra against an in vitro biofilm model [21] and its pulp tissue dissolving ability were studied [22], and in both studies the addition of surfactants optimized NaOCl properties in comparison to pure NaOCl.

Wettability is one of the most important physicochemical properties to a root canal irrigant. Contact angle measurements determine the wettability of the substratum. Low contact angles are an indication of good wetting, whereas high contact angles indicate poor wetting [25,26]. With the improvement of wettability, it is also possible that the irrigant could extend its solvent capability, and also optimize its bactericidal ability through a better penetration into the non instrumented areas of the root canal system [16]. Glantz and Hansson [14] reported that the contact between dentin and an irrigant depends on the wettability of the irrigant over the dentin surface in the root canal. Because spreading of the irrigant is thought to be directly related to its wetting properties, it may provide a good index to assess the irrigation efficiency in the root canal. The contact angle measurement on biological surfaces is usually difficult due to substrate hydration, porosity and heterogeneity. Dentin is a complex, heterogeneous and intrinsically wet organic tissue, composed by 50 vol% mineral, 30 vol% organic material, and 20 vol% fluid [27]. Besides its complex composition, dentin is penetrated by a tubular labyrinth containing odontoblastic cells. Tubule density and orientation vary from area to area [28].

The tubule lumen is lined by the peritubular dentin, which is highly mineralized. The tubules and peritubular dentin are separated by intertubular dentin. Dentin is hydrated in the vital state due to the pulp tissue pressure, estimated to be approximately 15 cm H₂O [29], which causes an outward flow of dentinal fluid. When dentin is instrumented or

grounded, a thin layer of debris, called *smear layer*, partially covers the surface, occluding the dentin tubules [30].

The contact angle as a thermodynamic equilibrium property of a liquid on a solid immersed in a fluid is unique only for inert, nonwetting liquids on ideal solids, for example, on smooth, homogeneous, inert, and nonporous surfaces [31,32].

In the present study, the limitation of the performed method should be carefully defined: the detected values were obtained on dentin, a nonideal surface (in the sense of the definition related to Young's equation). Dentin has a heterogeneous composition; it presents roughness and dentinal tubules [15,17–20]; it can present chemical interactions with some of the tested liquids. Consequently, the contact angle detected by placing a liquid droplet on the solid surface does not have the thermodynamic status of “equilibrium angle”, that is the three-phase boundary does not reside in the absolute minimum of the interface free energy. It is not possible and it is conceptually wrong, to link the measured values to surface free energy data, despite some existing examples in the relevant literature. It should be remarked, however, that the dentin surface was gridded and dry according to Stojicic et al. [22]. As a consequence, the tested surfaces are not completely comparable to the dentin surfaces found in clinical practice. Taking into account these limitations, the obtained data clearly demonstrated the effect of irrigation solutions on the wettability of dentin in vitro.

On dentin surface there is a range of metastable states, separated by free energy barriers [15,17–20]. All these “local minima” are accessible, and the liquid at the three-phase boundary can reside in one of the countless metastable states, depending on the height of the energy barriers and the macroscopic vibrational energy of the droplet. From a practical point of view, it means that a continuum of allowed values can be observed, across several degrees, all of them sharing the same thermodynamic status of metastable state. The most rigorous approach, as reported in the literature, involves the characterization of the wettability capacity through the measurement of the maximum and minimum allowed ranges, that is the advancing and receding contact angles [32]. The “sessile” contact angle is the contact angle detected by simply placing a drop on a surface. Even if not totally accurate, this method can be accepted in instances such as the present, when wide differences were detected among the tested liquids. The poor chemical-physical meaning of this value should, however, be considered. For example, 5.25% NaOCl solution could present contact angles across from 64 to zero, depending on its vibrational energy.

The contact angle of Chlor-Xtra on dentin reported by Stojicic et al. [22] was 36 degrees, while in the present study complete and immediate spreading was detected. This different results can easily be accounted for considering the different contributions to wetting on complex substrates like dentin. Generally, the observed contact angle contains a contribution from surface chemistry and a contribution from topography (capillary penetration). These two factors interact in a very complex way [17–19]. Probably, the preparation of the samples for measurement yielded different surface properties in both studies: contamination of the dentin surface during grinding and polishing can promote less wettable

surfaces. The grinding and polishing procedure can also yield different opening of the capillary structure of tubules, affecting the degree of capillary penetration. Present data show that capillary penetration is enhanced on present samples as compared to Stojcic et al. [22].

After defining the meaning of each detected value, it can be concluded that the high surface tension (71.4 mJ/m^2) observed in Group 4 indicated that MilliQ water does not penetrate into the porosity of the surface and shows a high contact angle. The detected value increases almost to 90 degrees before allowing the three-phase boundary to move. Pure 5.25% NaOCl showed an increased trend towards wetting as compared to water, possibly through the chemical interaction involving attack to organic tissue. On the other hand, the wettability of Hypoclean and Chlor-Xtra was completely controlled by capillary penetration. The combined effect of interface energetics and surface topography allows the liquid to readily penetrate into the dentin tubules and surface roughness.

Conclusions

Data clearly showed the superior wettability of sodium hypochlorite solutions modified with surfactants against dentin in comparison to 5.25% sodium hypochlorite and distilled water.

Clinical relevance: While having good dissolving tissue and antimicrobial capacities, sodium hypochlorite shows some limitations such as a high surface tension that limits its penetration in the anatomic irregularities of the root canal system and deeper penetration into dentineal tubules. The new solutions added with surfactants optimize the properties of sodium hypochlorite by lowering its surface tension. It is also possible that new irrigants could extend its solvent capability, and also optimize its bactericidal ability through a better penetration into the non instrumented areas of the root canal system.

Conflict of interest

The authors have no conflicts of interest to disclose.

Fundings

The authors declare that they have not received fundings for this study.

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at <http://dx.doi.org/10.1016/j.jgien.2012.06.002>.

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