Effect of Disinfectant Solutions on the Surface Free Energy and Wettability of Filling Material

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Abstract

Introduction: Gutta-percha (GP) and Resilon (Res) cones are usually disinfected by the use of solutions such as sodium hypochlorite (NaOCl) and chlorhexidine (CHX). The aims of this study were to evaluate the surface free energy of GP and Res cones after disinfection procedures and to investigate the wettability of endodontic sealers in contact with these surfaces. **Methods:** GP and Res flat smooth surfaces were prepared. Thirty-six samples of each material were used. The samples were divided into 6 groups as follows: Control group GP, immersed in Milli-Q water (MQW); CHX group GP, immersed in 2% CHX solution and then washed with MQW; NaOCl group GP, immersed in 5.25% NaOCl and then washed with MQW; Control group Res, immersed in MQW; CHX group Res, immersed in 2% CHX solution and then washed with MQW; and NaOCl group Res, immersed in 5.25% NaOCl and then washed with MQW. Samples were analyzed first with a goniometer to measure the contact angle between the test surfaces and 3 solutions (water, ethylene glycol, and diiodomethane) to determine the surface free energy. Then the contact angle between surfaces and each one of the sealers tested, AH Plus and Real Seal SE, was measured. Data were statistically analyzed. **Results:** The results showed that when disinfectant solutions were used, the surface free energy was greater. When evaluating the interaction between materials and sealers, the use of CHX presented lower values of contact angle, followed by NaOCl. **Conclusions:** It was concluded that the solutions used in decontamination increase the surface free energy, promoting high interaction between GP/Res and sealers. *(J Endod 2011;37:980–982)*

Key Words

AH Plus, chlorhexidine, cone disinfection, Real Seal SE, Resilon, sodium hypochlorite gutta-percha, surface free energy, wettability of filling material

The aim of obturation is to provide a three-dimensional seal of the root canal system, thus preventing bacteria ingress from the oral cavity and periradicular tissues. In addition, the filling materials might seal the root canal system and can be effective in entombing irritants that were not removed during preparation (1–3). Materials such as gutta-percha (GP) and Resilon (Res) cones, associated with sealers, are used for this purpose (4–6).

GP and Res cones are manufactured under aseptic conditions, but they can be contaminated by handling, aerosols, and physical sources during the storage process (7, 8). Because of their thermoplastic characteristics, the conventional heating processes cannot be used to sterilize them (3). Chemical substances such as sodium hypochlorite (NaOCl) and chlorhexidine (CHX) are, therefore, used for cone disinfection (9–11), but they alter the surface of these materials (12, 13).

The function of sealers is to fill root canal wall irregularities, apical ramifications, and deltas. In addition, the sealers act as a binding agent between root canal walls and the main filling material (GP or Res) by filling the spaces (14–16). The interface between either sealer and main filling material or sealer and dentin is, therefore, of prime clinical importance (17).

Adhesive materials must come into intimate contact with the substrate to facilitate molecular attraction and allow either chemical adhesion or penetration for micromechanical surface attachment. The adhesion processes are mainly influenced by the relative surface free energy (wetting ability) of the solid surface (18–20). The surface free energy is a measure of the surface reactivity or adhesiveness to its environment. This phenomenon occurs as a result of interatomic attraction. Measurement of surface free energy can be expressed in terms of the angle formed between a drop of liquid and the plane surface of the solid on which it rests. This is termed *contact angle*, and it has an inverse relationship with surface free energy (wettability), that is, the lower the contact angle, the greater the surface free energy and, hence, the greater the adhesion (21).

The physicochemical properties of a root canal sealer might characterize its clinical behavior during and after obturation of the root canal system (22, 23). One of these properties is the optimal wetting (24). The tendency of a liquid to spread on a solid surface is expressed with the formation of a contact angle (25). The surface with lower contact angle (greater surface free energy) presents high wettability, that is, in one solid with high surface free energy, the sealer spreads and interacts better with this surface, forming a low contact angle (17, 21).

The aims of this study were the following: (1) to evaluate the surface free energy of GP and Res cones after disinfection procedures and (2) to investigate the wettability of endodontic sealers in contact with these surfaces.

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TABLE 1. Values of Surface Free Energy for Different Experimental Groups

Groups	GP surface free energy (mean ± standard deviation), mJ/m^2	Res surface free energy (mean ± standard deviation), mJ/m^2
Water (control)	42.13 ± 3.21^c	41.54 ± 2.14^c
NaOCl	47.45 ± 2.15^b	44.28 ± 1.17^b
CHX	52.21 ± 2.17^a	51.53 ± 2.03^a

The superscript letters (^{a,b,c}) indicate, in each column, statistically significant values ($P < .05$).

Materials and Methods

Accessory GP (Odous De Deus, Belo Horizonte, MG, Brazil) and Res (Pentron Clinical Technologies, Wallingford, CT) cones of medium size were used.

The cones were placed between 2 glass plates on a heated surface to prepare flat smooth surfaces (5×5 mm). A total of 36 surfaces of each material were used.

All samples were washed with Milli-Q water (ultrapure water) and dried with nitrogen gas.

Measurement of Surface Free Energy

The samples were randomly divided according to the following experimental groups: Control group GP, GP immersed in Milli-Q water for 1 minute; CHX group GP, GP immersed in 2% CHX solution for 1 minute and then washed with 2 mL of Milli-Q water; NaOCl group GP, GP immersed in 5.25% NaOCl for 1 minute and then washed with 2 mL of Milli-Q water; Control group Res, Res immersed in Milli-Q water for 1 minute; CHX group Res, Res immersed in 2% CHX solution for 1 minute and then washed with 2 mL of Milli-Q water; and NaOCl group Res, Res immersed in 5.25% NaOCl for 1 minute and then washed with 2 mL of Milli-Q water.

After cleaning, the samples were dried with nitrogen gas and attached to a glass base with double-face tape.

The Ramé-hart goniometer (Ramé-hart Instrument Co, Netcong, NJ) was used to measure the contact angle between the experimental groups and the following solutions: water (polar), ethylene glycol (polar), and diiodomethane (apolar). In this step a total of 27 samples of each material were used, with 9 samples per group and 3 samples per solution. In each sample, 1 drop of liquid was measured. In each drop, 10 measurements were made. On the basis of the data regarding the 3 solutions above, the Ramé-hart software was able to measure the surface free energy of the different experimental groups.

Data were analyzed statistically by using Tukey and Mann-Whitney tests ($P < .05$).

Measurement of Contact Angle between Groups and Sealers

The same groups described above were used.

The Ramé-hart goniometer was used to measure the contact angle between AH Plus sealer (Dentsply, Petrópolis, Rio de Janeiro, Brazil) and GP groups (Control, CHX, and NaOCl) as well as between Real Seal SE sealer (SybronEndo, Orange, CA) and Res groups (Control, CHX, NaOCl). In this step a total of 9 samples of each material were used, with 3 samples per group. In each sample, 1 drop of sealer was measured. Also in each drop, 10 measurements were made. Data were statistically analyzed by using Tukey and Mann-Whitney tests ($P < .05$).

Results

Table 1 lists the values of surface free energy for different experimental groups. When disinfectant solutions were used, the surface free energy was greater. Comparing the groups, surfaces treated with CHX presented the highest values of surface free energy.

Table 2 lists the variation of contact angle values for different treatments. The surfaces treated with CHX presented the lowest values of contact angle, followed by NaOCl and water.

Discussion

The present study evaluated the surface free energy of GP and Res cones after disinfection procedures by using NaOCl and CHX for 1 minute and the wettability of endodontic sealers in contact with these surfaces. One minute was chosen because according to Royal et al (11), this time was enough to disinfect the cones.

The results of the present study showed that the use of NaOCl and CHX increased the surface free energy of GP and Res. This result suggests that the agents mentioned above lead to an increase in the wettability of the GP and Res surfaces, thereby interfering positively with the adhesion mechanism. This change can be due to chemical modifications on the surface of these materials caused by the action of these solutions. The reasons cannot be associated with physical change in the core material because according to Wenzel equation, the increase of roughness leads to a decrease of contact angle, and the endodontic literature showed that the use of these solutions leads to a reduction of the surface roughness of these materials (12, 13). Comparing the 2 solutions evaluated, CHX was the best disinfectant solution compared with NaOCl, that is, presented high values of surface free energy.

The GP/AH Plus and Res/Real Seal pairs were used because studies evaluating the interaction between these pairs for root canal filling are widely reported in the literature (26–28).

The contact angle is used to measure interaction between a solid surface and a liquid; in the present study these were the main filling

TABLE 2. Values of Contact Angle for Different Experimental Groups

Groups	GP/AH Plus (mean ± standard deviation), mJ/m^2	Res/Real Seal SE (mean ± standard deviation), mJ/m^2
Water (control)	68.3 ± 0.39^c	69.49 ± 0.12^c
NaOCl	58.49 ± 0.48^b	62.92 ± 0.13^b
CHX	48.29 ± 0.99^a	53.89 ± 0.8^a

The superscript letters (^{a,b,c}) indicate, in each column, statistically significant values ($P < .05$).

material and endodontic sealers. When the contact angle is less than 90 degrees, the liquid wets the substrate; if it is greater than 90 degrees, it is said to be nonwetting. A zero contact angle represents complete wetting. Low contact angle values are associated with a better interaction between solid surface and a liquid, in this case the sealer (17).

When the materials were subjected to disinfectant procedures, there was a decrease of the contact angle values, especially when CHX was used. Therefore, CHX increases the values of surface free energy and decreases the contact angle in relation to the sealers, thus allowing a better wettability. No other work in the literature has evaluated the effect of the disinfection substances in the wettability of GP and Res surfaces.

Attal et al (28) and Hu et al (29) have found that NaOCl increases the wettability of dentin surface. In contrast, Dogan Buzoglu et al (30) have demonstrated that NaOCl decreases the wettability of dentin surface. At this time, there is no report on the evaluation of the action of CHX in dentin by using the contact angle.

Under the experimental conditions of the present study, the results showed that the solutions used for cone disinfection caused an increased surface free energy, promoting high interaction between GP/Res and sealers when compared with the surfaces without disinfection treatment. Further studies will evaluate the wettability of CHX-treated dentin surface as well as the interaction between treated dentin and endodontic sealers.

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