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ORIGINAL ARTICLE

# Improvement of the efficacy of endodontic solvents by ultrasonic agitation

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## 1. Introduction

The success rates of endodontic treatment can reach levels from 86% to 98% (Abitbol et al., 2003). However, apical periodontitis may still persist or develop after treatment due to various factors, namely coronal leakage, caries or fractures, thus enabling reinfection by microorganisms of the oral cavity or proliferation of microorganisms in persisting endodontic infections (Siqueira 2001; Correia-Sousa et al., 2015; Ferreira et al., 2017).

Endodontic retreatment aims to reduce the bacterial load to a level that enables periapical healing. Nevertheless, its outcome is poor – about 70.9–83% (Torabinejad et al., 2009; Ng et al., 2011). The cleanliness of root canals cannot be accurately assessed through conventional periapical radiography or magnifying devices, but residual gutta-percha is systematically found in micro-CT scans after retreatment (Oltra et al., 2017). Chloroform and xylene have been widely used as endodontic solvents but concerns about their toxicity and potential carcinogenic effect led to seeking alternatives (Tamse et al., 1986; Metzger et al., 2000; Vajrabhaya et al., 2004; Magalhaes et al., 2007). Essential oils, like eucalyptol or orange oil, are one of the most common alternative groups of solvents used to enhance the dissolution or softening of gutta-percha. However, although they are considered less toxic, they are also reported as less powerful (Zaccaro Scelza et al., 2006; Faria-Junior et al., 2011; Martos et al., 2011). Studies show that, despite all the currently available technology, it is still not possible to achieve the complete removal of the potentially infected filling materials, which prevents total debridement and effective bacteria control (Alves et al., 2016; Keles et al., 2016; Rossi-Fedele and Ahmed, 2017). Although solvents have been indicated to prevent complications, such as ledges or perforations, in retreatment procedures, the literature reports that their use may hinder the cleaning of the root canal (Horvath et al., 2009). Retreatment can be carried out with or without solvents, but professionals often prefer to remove gutta-percha mainly with instrumentation, even though this may be a longer and less predictable procedure (Sae-Lim et al., 2000; Khalilak et al., 2013). Thus, endodontic solvents have almost fallen out of use. A new insight on endodontic retreatment seems to be necessary.

Ultrasonic agitation (UA) has been recently reported to improve the efficacy of organic solvents in sealer dissolution (Alzraikat et al., 2016; Ferreira et al., 2017). Nevertheless, little attention has been given to the specific action agitation might

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have on gutta-percha dissolution. The rationale of this investigation is that a better removal of filling materials, through dissolution, would improve retreatment's outcome. Therefore, there is still a need for a more effective and still safe protocol.

The purpose of this investigation was to qualitatively and quantitatively assess the effect of UA on improving the dissolution ability of tetrachloroethylene, eucalyptol and orange oil as compared to chloroform.

## 2. Materials and methods

### 2.1. Assessment of gutta-percha dissolution

Gutta-percha bars (DentQ, Toulouse, France) were used. The selected solvents were tetrachloroethylene (Septodont, Saint-Maur-des-Fosses, Cedex, France), eucalyptol (Dentaflux, Madrid, Spain) and orange oil (Citrol, Biodinamica, Madrid, Spain). Chloroform (Fisher Scientific, Loughborough, Leicestershire, UK) was used as control ($n = 5$). The protocol used is explained in more detail in a previous investigation (Ferreira et al., 2017). In short, standardized stainless-steel molds (7-mm diameter and 3-mm height) were fixed on stainless-steel blades and put on a heating plate (AREX Digital PRO, VELP Scientifica, Italy); then, molds were filled with gutta-percha bars, which were softened by the heat, and placed into an incubator (IKA KS 4000 ic Control IKA-Werke GmbH, Staufen, Germany) at 37 °C for 48 h. Before being immersed in the solvents, the samples were weighed three times on a digital analytical scale (AB204-S Mettler Toledo, USA) (initial weight, W0).

Half of the gutta-percha samples were submerged in 10 ml of the corresponding solvent for 2 or 5 min ($n = 5$ per solvent and time) without UA. The other half of the samples were submerged in an ultrasonic bath (RETSCH Solutions in Milling & Sieving, Haan, Germany) with 10 ml of the corresponding solvent, at a frequency of 30 kHz for 2 or 5 min ($n = 5$ per solvent and time). Afterward, all specimens were dipped in 10 ml of distilled water for 10 min before setting in the incubator at 37 °C. After 48 h, they were weighed again (post-immersion weight, Wf).

The gutta-percha dissolution was measured as a percentage, by the following equation (W0 = initial weight; Wf = final weight):

$$Dissolution\% = \frac{W0 - Wf}{W0} \times 100$$

The study design was planned to take into account the number of levels in each of the factors: time (two levels: 2 and 5 min), UA (two levels: with or without) and solvent (four levels: chloroform, tetrachloroethylene, eucalyptol and orange oil), and the samples were randomly assigned to each combination. Only one investigator handled the samples (immersion in the different solvents, with and without UA). The calculation of gutta-percha dissolution and the statistical analysis were performed by another investigator, who was blinded to the test groups.

### 2.2. Topographical analysis

A scanning electron microscopy (SEM) analysis was performed to compare the surface features changes after contact with the most and the least effective solvent (not considering chloroform – the control), so as to assess UA influence. Samples were observed in a Quanta 400FEG SEM (FEI, Hillsboro, OR) equipment, and each specimen was coated with gold/palladium using an SPI Sputter Coater (SPI Supplies, West Chester, PA).

### 2.3. Statistical analysis

The G*Power v3.1.9.2 program was used to determine an a priori sample size. The procedure used was ANOVA with fixed effects, main effects and interactions, using an alpha-type error of 0.05 with a power (1-ß) of 0.95 and sixteen groups, with an effect size of 0.4. This procedure indicated that five specimens per group were the ideal size.

Data were collected and preprocessed in an Excel spreadsheet for posterior statistical analysis in the IBM SPSS Statistics v25.0 (SPSS, Chicago, Illinois, EUA). Considering the nature of the variables, this study included a descriptive analysis of variables (including graphs methods and summary statistics in tables) and a comparative study using factorial ANOVA with replication, to evaluate the effect of the three factors – time, UA and solvent, the respective interactions between these, and their relationship to the solubility of gutta-percha (dependent variable). All the conditions for applying the ANOVA procedure were evaluated for the residuals: normality distribution centered in zero, homoscedasticity and independence. Statistically significant differences were considered for p values lower than a decision rule alpha = 0.05.

## 3. Results

### 3.1. Assessment of gutta-percha dissolution

Table 1 summarizes mean and standard deviation values of gutta-percha dissolution in different solvents, with and without UA for 2 min and 5 min.

The full ANOVA model presents an $r^2 = 0.95$. Registered p values associated with the testing effects in this model were lower than 0.05. The following parameters showed statistically significant differences regarding the mean dissolution of gutta-percha: immersion time, type of solvent used, UA, solvent-time interaction, agitation-time interaction, solvent-agitation interaction and solvent-agitation-time interaction. Using the Bonferroni post-hoc tests for multiple comparisons between solvents, results showed significant differences in the mean values of gutta-percha's dissolution. Namely, results revealed that the solvent ability of tetrachloroethylene was similar to chloroform, and that orange oil was the least effective. Taking into account the results and the estimate mean values, we suggest the following order for gutta-percha's mean dissolution in the solvents:

$$\mu_{\text{Chloroform}} \geq \mu_{\text{Tetrachloroethylene}} > \mu_{\text{Eucalyptol}} > \mu_{\text{orange oil}}$$

Furthermore, UA significantly improved the mean dissolution of gutta-percha with all solvents, approaching values of weight loss similar to or surpassing the control (chloroform) without UA. The immersion time boosted the effect of all the solvents.

**Table 1** Mean weight loss (± standard deviations) of gutta-percha, after being immersed in the different solvents for 2 min and 5 min, with and without ultrasonic agitation (UA).

| Solvents | | 2 min | | 5 min | | Global |
|---|---|---|---|---|---|---|
| | | Without UA | With UA | Without UA | With UA | |
| Chloroform | n | 5 | 5 | 5 | 5 | 20 |
| | mean ± SD | 3.80 ± 1.13 | 10.58 ± 0.49 | 2.68 ± 2.15 | 23.52 ± 2.21 | 10.17 ± 8.69[a] |
| Tetrachloroethylene | n | 5 | 5 | 5 | 5 | 20 |
| | mean ± SD | 3.86 ± 0.89 | 10.86 ± 1.32 | 5.12 ± 0.68 | 17.41 ± 4.68 | 9.32 ± 5.96[a] |
| Eucalyptol | n | 5 | 5 | 5 | 5 | 20 |
| | mean ± SD | 1.49 ± 0.21 | 5.03 ± 1.66 | 1.68 ± 0.55 | 7.04 ± 1.99 | 3.81 ± 2.69 |
| Orange oil | n | 5 | 5 | 5 | 5 | 20 |
| | mean ± SD | 0.00 | 2.19 ± 0.62 | 0.00 | 5.46 ± 1.23 | 1.91 ± 2.38 |

[a] Values with no statistically significant differences ($p > .05$) are indicated by lowercase letters.

*3.2. Topographical analysis*

SEM analysis was performed on the experimental samples with the most effective solvent (tetrachloroethylene) and the least effective (orange oil), with and without UA. The effect of both solvents was clearly intensified by the use of UA, as shown in Fig. 1. The surface feature changes enabled the qualitative assessment before and after UA.

## 4. Discussion

The purpose of this investigation was to qualitatively and quantitatively assess the effect of UA on improving the dissolution of gutta-percha by endodontic solvents. The methodology chosen was adapted from the ISO 6876:2012 standard to determine the solubility of the set sealer in water (ISO 2012). The gutta-percha brand (DentQ, Toulouse, France) used in this study is recommended for the warm gutta-percha filling technique and has also been used in similar investigations (Tanomaru-Filho et al., 2010; Faria-Junior et al., 2011; Jantarat et al., 2013). Although different brands of gutta-percha may present differences in solubility, most probably resulting from variations in the percentage of resin, they seem to follow a general pattern of solubility (Tamse et al., 1986; Metzger et al., 2000). Furthermore, the bars' presentation helps to achieve a more accurate filling of the ring molds and is often used with the methodology proposed

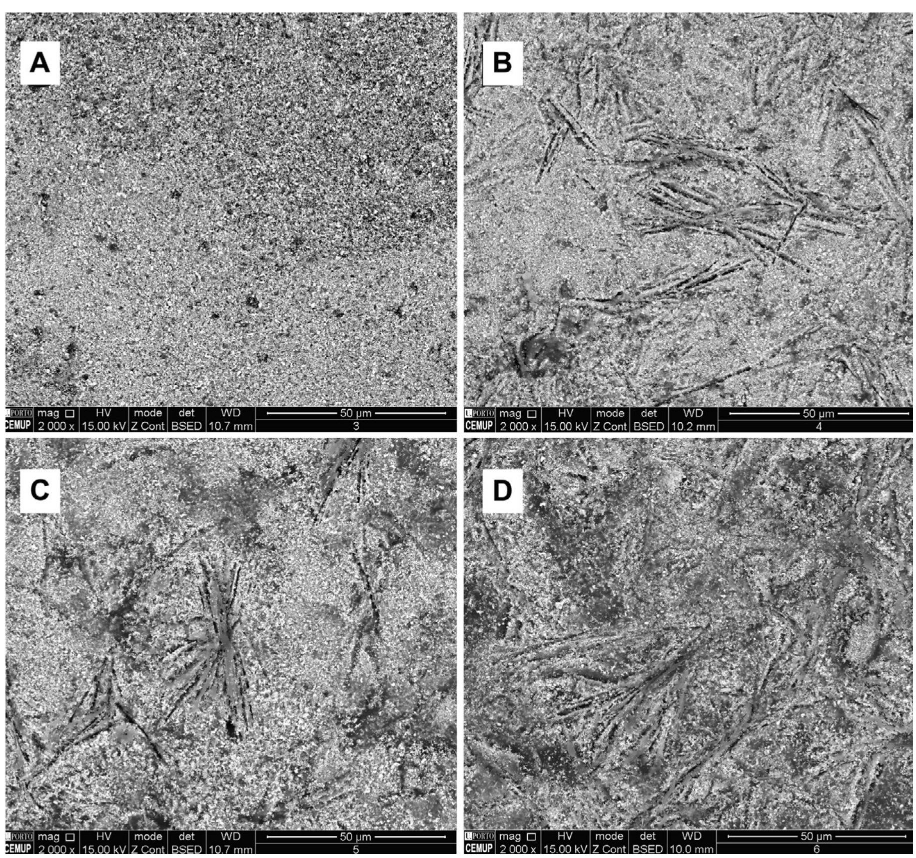


**Fig. 1** Representative SEM images at 2000× magnification. A – orange oil without UA; B – orange oil with UA; C – tetrachloroethylene without UA; and D – tetrachloroethylene with UA.

(Tanomaru-Filho et al., 2010; Faria-Junior et al., 2011; Jantarat et al., 2013).

The end-point measurements were assessed by the percentage of weight loss in two immersion periods, denoting the ability of the compounds to dissolve gutta-percha. They were firstly assessed without considering the mechanical approach (i.e., in the static environment), and the results clearly showed that tetrachloroethylene was the most effective and orange oil the least effective. This finding is corroborated by other investigations that considered tetrachloroethylene a safe alternative to chloroform (Tamse et al., 1986; Tanomaru-Filho et al., 2010; Faria-Junior et al., 2011). Contrarily, other authors have achieved a good efficacy with orange oil in gutta-percha, with a similar methodology and different gutta-percha presentation or considering softening as a measure of efficacy (Oyama et al., 2002; Jantarat et al., 2013).

Some investigators argued that solvents lead to more filling material remnants on the root canal walls and, thus, their use should be pondered only when it is difficult to reach the working length. It is important to mention the purpose of the solvent in retreatment (Horvath et al., 2009). If the expected solvent's effect is to enable gutta-percha removal with endodontic instruments, then softening with a milder solvent that has a slower evaporating rate is preferred (Metzger et al., 2000). This option would avoid messy procedures that leave residues adhered to the root canal walls. On the contrary, the purpose of the present proposal, with UA, is to remove small residues, often only microscopically detectable, after conventional retreatment instrumentation. For this purpose, an effective gutta-percha solvent, acting on hidden recesses through UA, without the potential hazards of chloroform, would fulfill the requirements.

The UA strategy dramatically improved the gutta-percha dissolution ability of all solvents. The rapid and continuous movement induced by ultrasound potentiates the solvents' dissolution ability while enhancing the displacement of the gutta-percha remnants adhered to canal walls, thus improving the percentage of filling material removed and, ultimately, the disinfection of root canals (van der Sluis et al., 2007). In these conditions, even orange oil, which was the least effective gutta-percha solvent, surpassed chloroform's static values and, thus, might become an effective and safer alternative to the traditional chloroform.

Similar to many other investigations, immersion time also boosted the effect of solvents, and this was even more relevant in UA groups (Alzraikat et al., 2016; Ferreira et al., 2017).

One of the limitations of the present methodology is that the results cannot be directly extrapolated to the in vivo situation. The method used, similar to other studies (Magalhaes et al., 2007; Faria-Junior et al., 2011; Alzraikat et al., 2016; Ferreira et al., 2017), was adopted due to its simplicity and reproducibility while enabling a greater standardization and a more accurate result assessment. It should be noted that factors such as bacterial load, the interaction between solvent and irrigation solutions, and solvent's volatility have not been considered. It is also clear that the contact area between solvent and gutta-percha in the stainless-steel rings is different from that observed in root canals and that radicular dentin/gutta-percha adhesion also differs from gutta-percha/stainless-steel ring adhesion. However, UA is expected to be even more effective in a clinic environment than in in vitro assays due to the ultrasonic vibration on the empty and wider canals, which occurs after filling materials' removal and reinstrumentation (Shrestha et al., 2009; Vivan et al., 2016).

The time, the renewal of the solvent solution and the agitation level are all factors that can contribute to the effectiveness of the treatment in the dental clinic. To our knowledge, this is one of the few reports about the effect of UA on solvents' ability to dissolve gutta-percha. Concerns about the risk of greater extrusion using solvents in retreatment seem unfounded (Vajrabhaya et al., 2004; Canakci et al., 2015; Keskin et al., 2017). The UA strategy here proposed is an alternative way of improving the dissolution ability by potentiating the solvents' action, thus avoiding stronger or more toxic proposals. With this strategy, less effective solvents can achieve good performances, reaching a balance between efficacy and safety.

SEM clearly highlighted the strong action of tetrachloroethylene potentiated by UA (Fig. 1. C and D). The changes on gutta-percha samples' surfaces after immersion in orange oil, although slight, support another important property of this solvent as a softener of gutta-percha, thus enabling an easier penetration of the instruments in the initial phase of retreatment procedures (Oyama et al., 2002; Jantarat et al., 2013). It is clinically relevant to understand the dissolution ability of each specific endodontic solvent in order to select them better for specific purposes (Rossi-Fedele and Ahmed, 2017).

Research should study new biocompatible solutions with clinical efficacy and a better delivery into dentinal tubules that improve the dissolution of filling materials, to select the best clinical protocol without potential hazards to the dental structure or toxicity to the host.

## 5. Conclusions

Despite its limitations, the present study showed that UA is beneficial, as it increases the dissolution ability of endodontic solvents on gutta-percha and, thus, may provide better disinfection, which might ultimately enable a more successful endodontic retreatment.

## Ethical statement

The authors declare that no experiments were performed on humans or animals for this study.

## Declaration of Competing Interest

The authors declared that there is no conflict of interest

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