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# Comparing the softening effect of three gutta-percha solvents on different types of gutta-percha with different application durations

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## ABSTRACT

**Introduction:** During non-surgical endodontic retreatment, gutta-percha (GP) solvents are indispensable in difficult cases when used with mechanical removal, however studies comparing their efficacy against different types of GP are limited. The purpose of this study was to investigate the softening effect of three solvents on the conventional (CGP), cross-linked carrier-based (CLGP), and thermoplasticized (TGP) and compare the effect of time on the softening effect of the solvents.

**Methods:** Tested GP were embedded in cuboidal blocks of stone with their upper surfaces exposed (1 mm diameter). Three commercial GP solvents based on D-Limonene (DL), Eucalyptol oil (EO), and orange oil (OO) were added to the exposed GP before an indenter (weight = 1Kg) was applied. Using a digital camera, the indentation depth was measured (mm) directly after applying the solvent and indenter (T = 0), and after 1, 2, and 3 min of application (T = 1,2,3). The means of indentation depth were calculated and compared using a two-way analysis of variance and Tukey's post-hoc test to assess the effect of the types of solvent, GP for each application duration, and Friedman's test to evaluate the effect of application duration on the softening effect of solvents.

**Results:** The type of GP (F = 261.43, p < 0.001), type of solvent (F = 3.57, p = 0.015), and application duration (F = 53.088, p < 0.001) were all found to significantly affect GP softening. DL exhibited the highest and only significant effect on CGP after 1 min (p < 0.05), while OO had the only significant effect against CLGP when applied for at least 2 min (p < 0.05). Both OO and EO had significant softening effects on TGP instantly or after 1 min of application, respectively.

**Conclusions:** The results of this study revealed that the softening effect of GP solvents varies depending on their type, their application duration, and the type of GP.

## 1. Introduction

Non-surgical endodontic retreatment aims to restore periapical tissue health after failed endodontic treatment. A poor-quality root filling with inadequate apical seal may allow re-infection of the root canal system, causing persistence or recurrence of the periapical disease (Wong et al., 2021). Hence, sufficient chemo-mechanical preparation is crucial, which involves the additional challenge of removing pre-existent root filling material. Insufficient removal of the root filling material can hinder disinfection and allow bacterial pathogens to thrive

by preventing irrigation solutions from reaching all parts of the canal system (Haapasalo et al., 2005).

During non-surgical endodontic re-treatment, chemical solvents are recommended for the removal of the root filling material (Duncan and Chong, 2008; Virdee and Thomas, 2017). Although solvents are used adjunctively with mechanical removal (Rossi-Fedele and Ahmed, 2017) in select cases (Horvath et al., 2009), they are indispensable in difficult cases where canals are curved and narrow or when the root filling material is resistant to manual removal (Dotto et al., 2021). Until its clinical use was restricted due to potential carcinogenic effects, chloroform was

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**Table 1**  
List of materials.

Gutta-percha	Conventional	Metabiomed, Cheongju, Chungcheongbuk, Republic of Korea
	Cross-linked carrier-based	GuttaCore®
	Thermoplasticized	Dentsply Sirona, Ballaigues, Switzerland Metabiomed, Cheongju, Chungcheongbuk, Republic of Korea
Solvents	D-limonene	Carvene Prevest DentPro, Jammu, India
	Eucalyptus oil	Eukalyptol CERKAMED, Stalowa Wola, Poland
	Orange oil	Orange Guttane CERKAMED, Stalowa Wola, Poland

the most effective and commonly used solvent (Barbosa et al., 1994; National Toxicology Program [NTP], 2021). Various chemical solvents, such as eucalyptus and orange oils, have been suggested as substitutes for chloroform (Schäfer and Zandbiglari, 2002; Whitworth and Boursin, 2000). These substitutes have been reported to have a reliability similar to that of chloroform (Good and McCammon, 2012; Hunter et al., 1991; Rehman et al., 2013) but not the same efficacy (Kazi et al., 2018). Thus, although the substitute solvents reduced the time required to instrument the full working length of the root canals, they did not show superior removal of the obturation material (Sağlam et al., 2014).

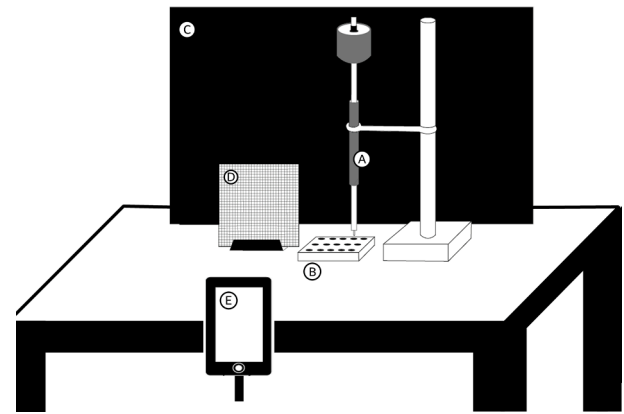
During obturation, gutta-percha (GP) is compacted into the prepared canals along with the sealer, either without heating or in the form of carrier-based or injectable thermoplasticized GP. While carrier-based thermoplasticized GP have used plastic carriers to deliver heat-softened GP, new carriers are made of cross-linked GP (GuttaCore®, Dentsply Sirona, Ballaigues, Switzerland), which is believed to provide easier retrievability (Beasley et al., 2013). Despite numerous studies on the softening effects of different GP solvents, there is limited evidence of their efficiency when used with cross-linked and other types of GP. To the best of our knowledge, no past study no previous study has evaluated the impact of solvents on various types of GP, including the recently introduced cross-linked GP (CLGP).

This in-vitro study aimed to evaluate the softening effect of three commercially available GP solvents (D-limonene (DL), eucalyptus oil (EO), and orange oil (OO)) on different types of GP—conventional (CGP), cross-linked carrier-based (CLGP), and thermoplasticized (TGP)—and evaluate the influence of time on the softening effect. The null hypothesis states that the type of GP, type of GP solvent, and the application duration have no effect on the softening capacity of GP solvents.

## 2. Materials and methods

### 2.1. Sample preparation

A total of 180 cylindrical GP samples (height: 5 mm, diameter: 1 mm) were prepared and embedded in stone. Three types of GP were tested (Table 1): conventional GP (CGP) (MetaBiomed, Cheongju, Chungcheongbuk, Republic of Korea), cross-linked carrier-based GP (CLGP) (GuttaCore®, Dentsply Sirona, Ballaigues, Switzerland), and thermoplasticized GP (TGP) (METABIOMED, Cheongju, Chungcheongbuk, Republic of Korea). Prior to GP embedding, freshly mixed stone was poured into a cuboid plastic mold with a removable cover containing 15 round holes of 1 mm diameter. Gutta-percha cones of CGP (size 80) were cut into 10 mm pieces and inserted vertically through the holes; these were used to fix the GP points while pouring the stone. For the CLGP, 10 mm of the carrier (size 30, diameter 1 mm) was cut just below the handle and embedded in the stone. The same steps were followed for the TGP group, but the CGP cones were additionally coated with petroleum jelly to ease removal before injecting the TGP into the spaces. The CGP cones were removed after the stone was set, and the TGP was injected into the cylindrical spaces left using a B&L Beta device (B&L Biotech, VA, USA) and



**Fig. 1.** The indentation testing apparatus. (A) Metal rod with a pointed tip attached to a holder that allows vertical movement only. (B) Gutta-percha embedded in cuboidal blocks of stone with their surfaces exposed. (C) Black board for photography background. (D) Graph paper (1 mm) as a reference to measure the indentation depth. (E) Mobile phone digital camera mounted at the same level as the upper surface of the stone block.

compacted using a size 4 Machtou hand plugger (Dentsply Sirona, Ballaigues, Switzerland). After setting, the molds were dismantled, excess GP was cut, and both surfaces of the stone block were carefully ground flat using 400-grit silicon carbide polishing paper. Three different GP solvents were tested: DL-based (Carvene, PREVEST DenPro, India), EO-based (Cerkamed, Stalowa Wola, Poland), and OO-based (Cerkamed, Stalowa Wola, Poland). In an additional control group, distilled water (W) was used instead of the solvent. To test the softening effect of the solvents, an in-house-made indenter was used (Fig. 1). The stainless-steel indenter's tip had a diameter of 0.15 mm with 15° of angulation and was attached to a fixture to allow vertical displacement only. A stainless-steel weight was added to the upper end of the indenter, making its total weight 1020 gm, which is equivalent to 10 N force (Hunter et al., 1991). Before testing, a drop (0.25 mL) of the tested solvent was applied to the surface of the exposed GP using plastic transfer pipettes, and then the indenter was placed on the middle of the exposed GP surface of each sample. A digital photograph was obtained using a 12.0 MP mobile phone camera (iPhone12, Apple Inc., CA, USA) mounted level with the block's upper surface in a standardized position. Photographs were taken directly upon application of the indenter ( $T = 0$ ) and after 1 ( $T = 1$ ), 2 ( $T = 2$ ), and 3 ( $T = 3$ ) minutes. Photographs were uploaded to the image processing software ImageJ 1.53v (National Institutes of Health, Bethesda, MD, USA) to facilitate measurement of the depth at which the indenter's tip penetrated the GP. The measuring tool of the software was calibrated to 1 mm graph paper mounted next to the indenter. All photographs were taken with a black background under consistent room lighting. The indentation depth of the indenter was measured in millimeters (mm) as the difference between the original length ( $L$ ) of the indenter's tip and the length of the tip measured after application ( $L_x$ ).

$$\text{Indentation depth (mm)} = L - L_x$$

### 2.2. Statistical analysis

The data were entered into IBM-SPSS for Windows 28.0 software (SPSS Inc., Chicago, IL). Continuous data were described using measures of tendency and dispersion. The normality of continuous variables (measurements at  $T = 0$  to  $T = 3$ ) was tested using the Shapiro–Wilk test. To assess the effect of the type of GP and GP solvent on the indentation depth at each application duration, a two-way analysis of variance (ANOVA) was performed followed by a post-hoc Tukey's test. Friedman's test of nonparametric repeated measures was used to compare the effects of different application durations of the GP solvents on the

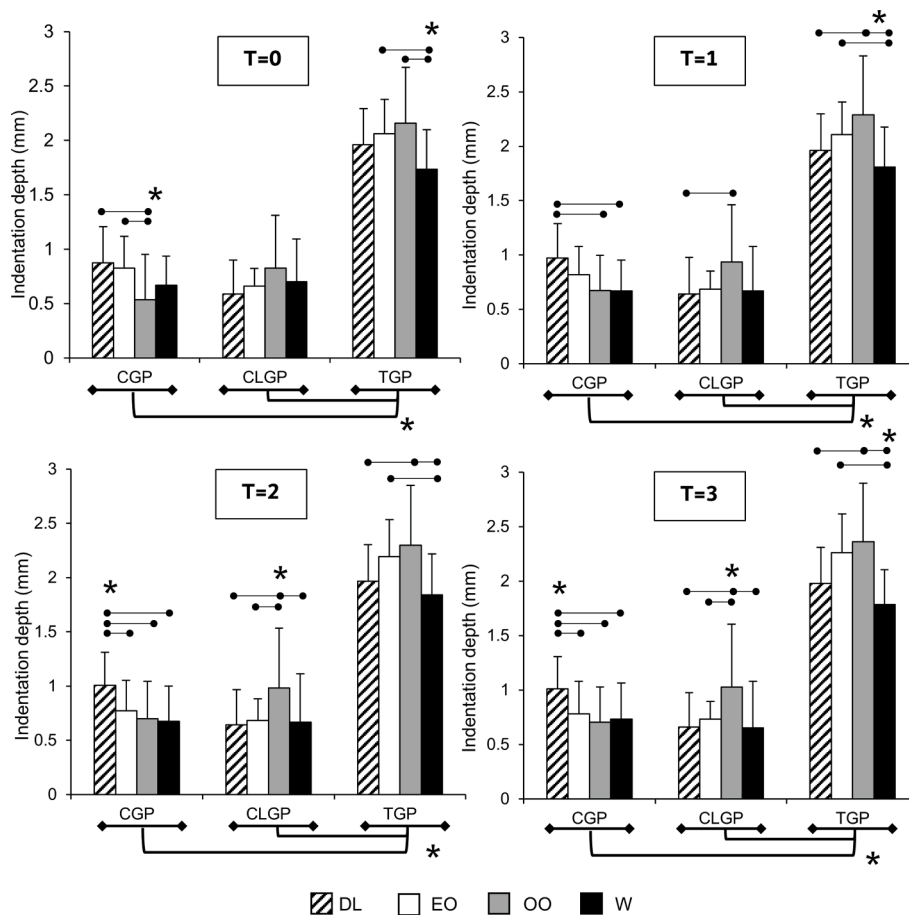


Fig. 2. Indentation depth measurements representing the softening effect of each GP solvent (DL, EO, OO) on different types of Gutta-percha (CGP, CLGP, TGP) in comparison to water (W) at different application durations (T = 0,1, 2, 3 min). Based on two-way ANOVA and Tukey’s post-hoc tests, statistically significant differences ( $p < 0.05$ ) are indicated with (\*).

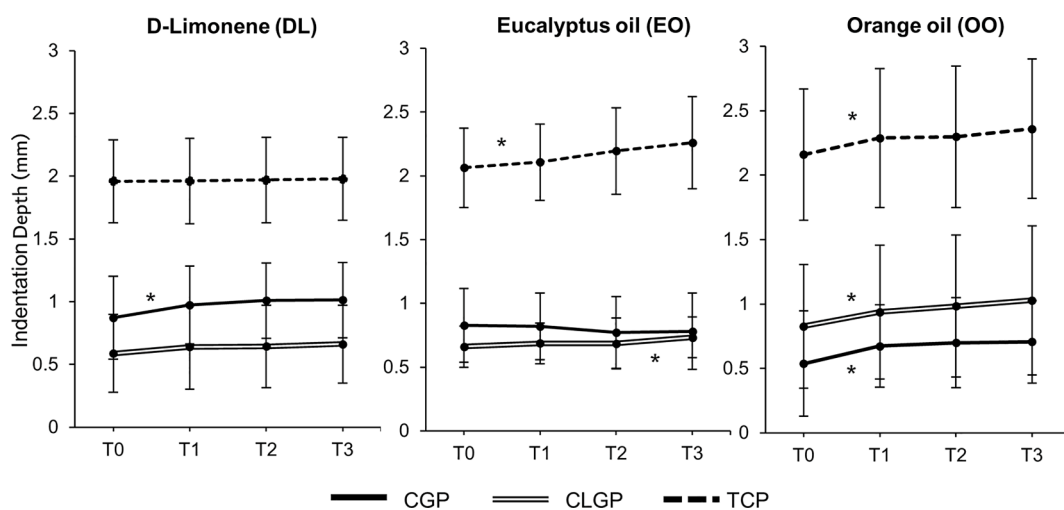


Fig. 3. The effect of time on the indentation depth representing the softening effect of each solvent (DL, EO, OO) on different types of Gutta-percha (CGP, CLGP, TGP). Based on Friedman’s test, (\*) Indicates statistically significant differences between the consequent data points ( $p < 0.05$ ).

different types of GP. A p-value of less than 0.05 was considered significant in all statistical analyses.

### 3. Results

The indentation depth measurements of each type of GP with each

solvent at all time points (T = 0, T = 1, T = 2, and T = 3 min) are presented in Fig. 2. At each time point, the softening effect varied depending on the GP type and the solvent used based on the two-way ANOVA analysis. Comparing the indentation depth of different GP types showed TGP to be associated with the greatest values regardless of the duration or type of solvent used and to be significantly greater than

those of CGP and CLGP at all time intervals ( $P < 0.05$ ).

The greatest softening effect on CGP was associated with DL at all time points, which was significantly more than the control and OO groups after 1 min and EO after 2 min (Fig. 2). Compared to the other solvents, DL was the only solvent associated with a significantly greater softening effect on CGP than water. For CLGP, however, the greatest softening effect was associated with OO at all time points, which was significantly higher than that of the control (W) and the other two solvents after 2 and 3 min. The TGP was most affected by OO and EO, in which significantly deeper indentation occurred at  $T = 0$  and  $T = 1$ , respectively, when compared to W. There was no significant difference in the softening effects of W or DL on TGP at all time points.

The effect of time on the softening effect of each solvent based on Friedman's test of nonparametric repeated measures is illustrated in Fig. 3. The DL was found to have significantly improved softening effect on CGP after 1 min of application, with no similar effect of time seen when it was applied to CLGP or TGP. For EO, the softening effect improved significantly after 1 min application on TCP and after 3 min application on CLGP only. The OO exhibited significant improvement in its softening effect after 1 min of application on all types of GP.

#### 4. Discussion

This study compared the efficacy of three commercially available solvents in softening different types of GP and evaluated the effect of time on their efficacy. Based on our results, the null hypothesis can be rejected, as the different types of GP varied in their response to the softening effect of the same solvents (Fig. 2). Additionally, GP solvents were found to vary in their softening effects, depending on the type of GP and the duration of the application (Fig. 3).

In terms of the effect of different solvents on CGP, DL was demonstrated to have the best softening effect, requiring only 1 min (Fig. 3). Conversely, the OO and EO did not have greater effects than water, regardless of the duration of application, which is a result in agreement with a previous study (Tanomaru-Filho et al., 2010). In their study, de Oliveira et al. (2017) reported that EO, when used with CGP, had the least effective dissolution ability when compared to OO, xylol, and citrol. However, their results contradict those of previous studies on CGP that reported these oils to have comparable effects to that of chloroform (Rehman et al., 2013) and higher dissolving abilities than that of water (Magalhães et al., 2007; Oyama et al., 2002; Tanomaru-Filho et al., 2010). Our results did not show significant differences between the effects of OO and EO on CGP, which, despite disagreement about their efficacy on CGP, agrees with the results of previous studies (de Almeida Gomes et al., 2013).

On CLGP, OO was the only solvent that exhibited significantly deeper penetration than the negative control (water) when applied for 2 min or longer (Fig. 2). This provides evidence about the ability of this solvent to soften carriers manufactured from cross-linked GP; DL and EO did not exhibit the same capacity. On TGP, both OO and EO exhibited better softening effects than DL, in agreement with Tanomaru-Filho et al. (2010). Eucalyptol was found to be more effective on TGP than on CGP and CLGP, which is in agreement with a previous study by Dagna et al. (2017), who reported EO to have an effect comparable to that of chloroform on TGP. This contrasts with another study by Faria-Júnior et al. (de Faria-Júnior et al., 2011) who showed comparable effect of EO against TGP and CGP. In the present study, similar to EO, OO was found to be efficient when used on TGP, which is in agreement with the results of Hansen et al. (1998), and to have a unique softening effect on CLGP.

Although both the OO and DL used in this study were chemically relevant and were obtained from the same source, the former was 100 % orange oil while the latter was composed of D-limonene (<10 %) and mineral oil (90 %) (Cerkamed Medical Company, n.d.; Prevest Denpro, n.d.). This may explain the differences found in the efficacy of these solvents against different types of GP. The variation in the chemical composition of these solvents might have affected their capacity to

soften the GP, which was confirmed by our findings that different solvents were associated with different indentation depths for the same type of GP. For all solvents, the softening effect was most evident on the TGP. This can be attributed to the composition of TGP compared to that of CGP, with a lower percentage of inorganic components rendering TGP softer (El-Hawary et al., 2015; Maniglia-Ferreira et al., 2013). This explains the higher penetration values found in testing TGP even when water was used. Hence, comparing the softening effects of the solvents with a negative control (water) in the same GP was essential to verify the results and overcome variations among the GP types tested. Such variations in GP composition and its impact on the softening effect of solvents should be taken into consideration when testing their efficacy in vitro. Testing different types of GP is particularly important because clinicians are often unable to identify the type of root filling material they are dealing with.

The indentation test applied in this study has been used previously to assess the softening effects of solvents on GP (Hunter et al., 1991; Wennberg and Ørstavik, 1989). Such tests may be more clinically relevant than those assessing changes in GP mass after immersion in solvents. The process in the current study allowed for the evaluation of the actual softening effect of solvents over time rather than only assessing their dissolving capacity. Our findings confirmed that the duration of application of the three tested solvents affected their softening capacity, which is in agreement with the results of previous (Martos et al., 2011; Rubino et al., 2012). One factor that should be considered, however, is the ability of the solvent to penetrate deeper parts of the GP, including those beneath the indenter. Without the ability for significant penetration, the solvent might not reach the deepest points necessary to soften the GP, which may restrict its effectiveness.

From a clinical point of view, the use of manual filing remains the most efficient and safest approach to removing root filling materials in a retreatment procedure (Dotto et al., 2021). However, under certain circumstances, using GP solvents may ease this process and facilitate access to the full length of the root canal (Horvath et al., 2009). Based on the findings of this study, clinicians should consider the use of different solvents when attempting to remove a root filling, knowing that they are unable to identify the nature of the root filling used in the previous treatment. Additionally, regardless of the type of solvent used, sufficient time—not less than 1 min—should be given before flushing the solvents from the canal. Considering its wider softening capacity on different types of GP, OO-based solvents seem to be the preferable starting option.

#### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

#### Appendix A. Supplementary material

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.sdentj.2023.10.025>.

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