Softening Efficacy of Various Solvents on Gutta-percha and Root Canal Sealer

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ABSTRACT
Solvents have been used in endodontic retreatment for a long time and the dissolving effect is well proven. Latterly chloroform has come in a bad light due to its possible carcinogenicity. Despite the negative health effect it is still used in dental environment. Other more biocompatible solvents have now reached the market and tests should be performed to evaluate the softening efficacy. The purpose of this in-vitro study was to evaluate the softening efficacy of four different solvents used in endodontics; chloroform, eucalyptol, tetrachloroethylene, orange-oil and a control group. 100 simulated canals filled with gutta-percha, epoxy amine resin based sealer, zinc oxide eugenol based sealer and non eugenol calcium hydroxide based sealer were tested with hardness measurement before and after two minutes exposure time of medicament. Non-eugenol calcium hydroxide failed to set and was excluded from the test. A Shore A durometer was used to evaluate the hardness of the materials and all data was first analyzed with Kruskal-Wallis test and then Mann-Whitney test to compare with control group. With a digital camera connected to a microscope we also took pictures to compare the impressions with the different medicaments. The result showed that chloroform and tetrachloroethylene is significant better to soften gutta-percha than control group (p < 0.05) but only chloroform significant better than control group to soften epoxy amine resin based sealer and zinc oxide eugenol based sealer (p < 0.05). Even if the result was significant, we cannot draw any conclusions due to small sample size.
INTRODUCTION

The rate of success in endodontic treatments is between 62% and 96% (Sjögren et al., 1990; Friedman et al., 2003). That means that, in a certain number of cases, there is a need of retreatment. In the procedure of retreatment all root filling material in the pulp has to be removed in order to disinfect the canals. Mechanical instruments can be used but the use of these instruments induces a risk of fractures and perforations (Souter and Messer, 2005). The shape of the canal can also make it difficult to use only mechanical instruments (Bodrumlu et al., 2008). Complete removal of filling material from the root canals, independent of the retreatment technique and sealer type, cannot be achieved (Kosti et al., 2006). The apical third contains significantly more residuary filling material than the coronal part (Ersev et al., 2012). To ease the struggle of complete removal of all filling material, a combination of conventional mechanical cleaning and dissolving liquids will allow a higher degree of apical third cleansing (Bodrumlu et al., 2008).

Gutta-percha was proposed as a dental material already in 1833 (Pecora et al., 1993). It is a natural thermoplastic material and is used for its biocompatibility and melting and moldability properties (Ørstavik, 2008). Nowadays different types of sealers are used in combination with gutta-percha as a root filling method. The properties of sealers are the sealing and antimicrobial effect (Seelan et al., 2015).

Chloroform, eucalyptol and orange oil are solvents often used for removing of root filling material (Karatas et al., 2015). Chloroform has been used as a solvent for gutta-percha since 1910 and the dissolving effect of chloroform is well proven (Pecora et al., 1993). Even though, it is highly toxic and possibly carcinogenic (International Agency for Research on Cancer, 2014), the usage during endodontic treatment is not harmful for patients if used within adequate dose levels (Chutich et al., 1998). Due to the carcinogenic effects of chloroform, other solvents has been developed and tested for clinical usage. Orange oil is an essential oil recovered from the peel of orange, *Citrus aurantium*, and was introduced into dentistry in 1975 (Roselino et al., 1975; Magalhães et al., 2007). Another alternative to chloroform is eucalyptol, which is a cyclic ether and monoterpenoid. It is a colourless liquid made from natural organic compound and is a usual ingredient in mouthwash and cough suppressant (The Drugbank database, 2013).
Furthermore, tetrachloroethylene is also a solvent that is available for the dentistry in Sweden. It is classified as a group 2A, which means probably carcinogenic to humans, by International Agency for Research on Cancer (IARC) in 2014.

The purpose of this study was to evaluate the softening efficacy of four different solvents on gutta-percha and sealer. With a Shore A hardness test we measured the hardness of gutta-percha and three different types of sealer before and after exposure of the solvents.

**MATERIAL AND METHOD**

**Ethical considerations**
The Ethics Forum at the Department of Odontology, Umeå University finds that no appropriate ethical considerations have been integrated into this study. All tested medicaments were purchased at the Swedish dental market. In addition, the medicaments got CE markings which declare that the product meets the requirements of the applicable European Union directives.

**Literature search**
The PubMed database (U.S National Library of medicine, USA) was used to search for articles. The MeSH terms *endodontic treatment*, *endodontic retreatment*, *gutta-percha solvent*, *chloroform endodontic*, *sealer antimicrobial*, *shore hardness*. Further articles were found when reviewing reference lists from other studies. Free text search was carried out from Google (Google, Inc., USA).

**Study design**
Experimental study.

**Testing procedure**
100 canals were prepared in plastic blocks and divided into four groups of 25. The canals were prepared with a pillar drill (⌀ 3.5 mm, 7 mm in depth) and each group was either filled with gutta-percha, epoxy amine resin based sealer, zinc-oxide eugenol based sealer or non-eugenol calcium hydroxide based sealer. One commercial brand of gutta-percha was used (DeTrey, Dentsply, York, PA, USA). Three types of sealer were used; one epoxy amine resin based (AH-Plus, Dentsply, York, PA, USA), one zinc-oxide eugenol based (Tubliseal, Kerr,
Los Angeles, CA, USA) and one non-eugenol calcium hydroxide based (Sealapex, Kerr, Los Angeles, CA, USA). To allow setting of the sealers the specimens were stored in 100% humidity (37°C) and kept under these conditions for three days. Sealapex failed to set and was not included in the test. An industrial turret-milling machine was used to smooth the sides of the blocks to be parallel and to flatten the surface of the test materials.

The untreated test materials were measured in a Shore A durometer type (Shore, Durotronic model 1000, USA) as a start value. Each group of gutta-percha or sealer were then divided into subgroups of five according to the medicaments, which is displayed by figure 2 in the appendix. The tested medicaments were tetrachloroethylene (Endosolv: Septodont, Saint-Maur-des-Fossés, France), eucalyptol (Guttasolv: Septodont, Saint-Maur-des-Fossés, France), orange oil (GP-solvent: Prevest Denpro, Heidelberg, Germany), chloroform (Labservice AB, Sundsvall, Sweden) and sterilised water as control. With a pipette the specimens were exposed to 10 μL of the different medicaments for two minutes and then measured again. In order to reduce evaporation a model of dental putty with a prepared hole for each specimen was made. In addition to reduce evaporation this model also acted as a reservoir and kept the test medicament centred on the test material.

To assess presence of impressions in the material after medicament treatment a microscope was used and pictures of impressions were taken with a digital camera connected to the microscope.

**Data management**

IBM SPSS Software was used to test if there were significant differences between the solvents for each of the three materials. First a Kruskal-Wallis test (p < 0.05) was used to see if there was a statistically significant difference between the tested solvents. Then, a Mann-Whitney test (p < 0.05) was used to see differences for each solvent in compare to control.

**RESULTS**

The results of this *in vitro* study are presented in Table 1. Tetrachloroethylene and chloroform showed statistically significant differences in softening gutta-percha compared to control (p < 0.05). Chloroform showed statistically significant differences on both AH-plus and Tübliiseal (p < 0.05). Eucalyptol and orange oil showed no statistically significant differences in softening gutta-percha (p > 0.05) and tetrachloroethylene, eucalyptol and orange-oil showed no statistically significant differences on AH-Plus and Tübliiseal (p >
The photos taken with microscope clearly showed impressions in the test materials that were exposed to tetrachloroethylene and chloroform.

DISCUSSION
The objective of this study was to evaluate the ability of a solvent to soften the gutta-percha and sealer. According to the result in this experimental study, chloroform and tetrachloroethylene indicates a significant softening effect on gutta-percha. Chloroform also exhibit significant effect on both tested sealers but tetrachloroethylene only shows marginal effect.

Previous studies show various results of these solvents but the consensus is that they almost have the same dissolving effect with some small differences. On gutta-percha, one study shows similar effect for chloroform, orange oil and eucalyptol and other studies show similar effect for chloroform and eucalyptol (Magalhães et al., 2007; Hunter et al., 1991). In contrast, one study shows no statistically difference between chloroform and orange oil but they both were statistically better than eucalyptol (Oyama et al., 2002). On sealer, some studies indicate similar result as the present study (Bayram et al., 2015; Whitworth & Boursin, 2000; Schäfer & Zandbiglari, 2002). Other studies indicate that chloroform, eucalyptol and orange oil have the same dissolving effect on sealers (Ring et al., 2009; Bodrumlu et al., 2008; Scelza et al., 2007).

The discrepancy in results may be the difference in execution. In one study, as in the present study, specimens were used in combination with pressure measurement (Oyama et al., 2002). However, in most studies they compare weight loss of the specimens before and after exposure of solvent. In a study, extracted teeth were used in combination with electron microscope to indicate dissolving efficacy (Ring et al., 2009). By using Shore A measurement, a standardized amount of pressure could be used. Another benefit was the narrow tip that can resemble to endodontic files and Gates drill. The disadvantage with this hardness method is that it is made for testing elastic materials instead we got impressions in the tested materials. This means that we can not compare our hardness values to elastic materials with similar values, but only compare our values to each other. Another difference is the exposure time. Previous studies have used exposure times lasting up to ten minutes (Bayram et al., 2015; Martos et al., 2011). Even though a shorter time of exposure, as used in this study, is more adequate to clinical usage. In order to reduce the clinical treatment time,
an early initiating dissolving effect is necessary for keeping efficiency of retreatment. The discrepancy in results may also be due to amount of solvent. Previous studies have used a larger amount of solvents than in the present study (Karatas et al., 2015; Scelza et al., 2007). The incitement of applying a minimal amount of solvent is to reduce the risk of exposing patients for potential carcinogenic or toxic products. Thus, if appropriate dose level is applied the carcinogenic and toxic exposure for patient and operator will decrease (Chutich et al., 1998).

During our pilot test we noticed that some of the medicaments evaporated almost instantaneously. Particularly chloroform and tetrachloroethylene evaporated rapidly due to their high volatility. In order to prevent evaporation a model of dental putty with prepared holes were made. Both chloroform and tetrachloroethylene have low surface tension which makes it complicated to keep it fixed on the test material. The model of dental putty eased this problem as well.

Sealapex failed to set unless in contact with air. Only the top layer was solid, and once the tip of the durometer penetrated the surface, the material underneath was unset. It has previously been showed difficulties with getting Sealapex to solidify (Erdemir et al., 2003).

Degree of toxicity should be a great factor when determining which solvent to select in the clinical environment. Even if small amount of the body is exposed, overfill and lack of handling the solvents are things to be reckoned with. Chloroform and eucalyptol are cytotoxic and it is considered a secondary mechanism in non-genotoxic carcinogens (Ribeiro et al., 2006). Orange oil is less cytotoxic than both chloroform and eucalyptol and therefore a better alternative in a degree of toxicity (Scelza et al., 2006). Tetrachlorethylene is both cytotoxic and mutagenic and can contribute an increased risk of getting lung cancer and possibly colon-rectum cancer but it is not tested in dental environment and how dose levels can be a factor (Paulu et al., 1999). Because of this, an important standpoint that all clinicians need to take, is to select medicaments which are highly efficient of dissolving residuary root canal filling material while poses no harm to the patient or to the therapist. Moreover, therapists should consider the choice of material in the initial endodontic treatments since the adversity of removing some sealer types, as present study and several others encountered.
CONCLUSION
Within the limitations of this *in vitro* study the following conclusions were made: both chloroform and tetrachloroethylene softened gutta-percha significantly better than control, orange oil and eucalyptol could not soften any of the tested materials and only chloroform were able to soften Tubliseal within the given timeframe. Orange oil, which is the most biocompatible solvent used in present study, showed no softening effect on any of the tested specimens and therefore the usage in endodontic retreatments is irrelevant. The finite amount of material may have influenced the result, even though it was significant. Further studies, with an extended amount of material, are required to establish the results.

ACKNOWLEDGMENTS
We are grateful for the help and guidance throughout this study from our tutor Malin Brundin. We would also like to thank Anna-Karin Hulterström for her expertise regarding measuring with the Shore A durometer. Finally, we are thankful for the materials and medicaments which were supported by the Department of Odontology.
REFERENCES


Table 1. Mean indicates the average decrease in Shore A-hardness of the materials before and after application of the medicaments. The value of statistically significance was set to 0.05 and control is used as reference.

<table>
<thead>
<tr>
<th>Material</th>
<th>Solvent</th>
<th>Mean</th>
<th>Std. Deviation</th>
<th>P-value(^a)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gutta-percha</td>
<td>Tetrachloroethylene</td>
<td>5.50</td>
<td>± 0.92</td>
<td>0.008</td>
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<td></td>
<td>Eucalyptol</td>
<td>0.36</td>
<td>± 0.38</td>
<td>0.151</td>
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<td></td>
<td>Orange oil</td>
<td>-0.04</td>
<td>± 0.39</td>
<td>0.690</td>
</tr>
<tr>
<td></td>
<td>Chloroform</td>
<td>5.44</td>
<td>± 1.59</td>
<td>0.008</td>
</tr>
<tr>
<td></td>
<td>Control</td>
<td>0.06</td>
<td>± 0.18</td>
<td>Ref</td>
</tr>
<tr>
<td>AH-Plus</td>
<td>Tetrachloroethylene</td>
<td>0.60</td>
<td>± 0.54</td>
<td>0.421</td>
</tr>
<tr>
<td></td>
<td>Eucalyptol</td>
<td>0.10</td>
<td>± 0.07</td>
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<tr>
<td></td>
<td>Orange oil</td>
<td>0.34</td>
<td>± 0.25</td>
<td>0.548</td>
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<tr>
<td></td>
<td>Chloroform</td>
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<td>± 0.93</td>
<td>0.008</td>
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<td></td>
<td>Control</td>
<td>0.28</td>
<td>± 0.35</td>
<td>Ref</td>
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<tr>
<td>Tubliseal</td>
<td>Tetrachloroethylene</td>
<td>0.76</td>
<td>± 0.77</td>
<td>0.056</td>
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<tr>
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<td>Eucalyptol</td>
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<td>± 0.27</td>
<td>0.421</td>
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<td></td>
<td>Orange oil</td>
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<tr>
<td></td>
<td>Chloroform</td>
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<td>± 0.90</td>
<td>0.008</td>
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<tr>
<td></td>
<td>Control</td>
<td>0.00</td>
<td>± 0.16</td>
<td>Ref</td>
</tr>
</tbody>
</table>

\(^a\) Mann-Whitney test
Table 2. A digital camera connected to a microscope collected following pictures of the impressions in the material after exposure to medicaments.

<table>
<thead>
<tr>
<th></th>
<th>AH-Plus</th>
<th>Tubliseal</th>
<th>Gutta-percha</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tetrachloroethylene</td>
<td><img src="Image1" alt="Image" /></td>
<td><img src="Image2" alt="Image" /></td>
<td><img src="Image3" alt="Image" /></td>
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<td>Eucalyptol</td>
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<td><img src="Image5" alt="Image" /></td>
<td><img src="Image6" alt="Image" /></td>
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<tr>
<td>Orange oil</td>
<td><img src="Image7" alt="Image" /></td>
<td><img src="Image8" alt="Image" /></td>
<td><img src="Image9" alt="Image" /></td>
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<tr>
<td>Chloroform</td>
<td><img src="Image10" alt="Image" /></td>
<td><img src="Image11" alt="Image" /></td>
<td><img src="Image12" alt="Image" /></td>
</tr>
<tr>
<td>Control</td>
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<td><img src="Image14" alt="Image" /></td>
<td><img src="Image15" alt="Image" /></td>
</tr>
</tbody>
</table>
Figure 1: Shore A durometer, used for measuring.
Figure 2. The figure illustrates the distribution of the specimens.