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Dissolving efficacy of eucalyptus and orange oil, xylol and chloroform solvents on different root canal sealers

J. Martos[1], A. P. S. Bassotto[2], M. P. González-Rodríguez[3] & C. M. Ferrer-Luque[3]

[1]Department of Semiology and Clinic, Faculty of Dentistry, Federal University of Pelotas, Pelotas, RS; [2]Private Practice, Caxias do Sul, RS; and [3]Department of Dental Pathology and Therapeutics, School of Dentistry, University of Granada, Spain

Abstract

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Aim To evaluate the solubility of five root canal sealers in orange oil, eucalyptol, xylol and chloroform solvents.

Methodology The solubility of RoekoSeal, Sealer 26, Epiphany, Endomethasone and EZ-Fill sealers was assessed in orange oil, eucalyptol, xylol, chloroform and distilled water. Seventy-five samples of root canal sealers were prepared and then divided into five groups for immersion in solvent for 2, 5 or 10 min. The means of loss weight were determined for each material in each solvent at all immersion periods, and the values were compared by factorial analysis of variance (ANOVA) and SNK multiple comparisons.

Results In the orange and eucalyptus oil groups, there was no significant difference among RoekoSeal, Sealer26, Epiphany and EZ-Fill at the three immersion periods ($P > 0.05$). With xylol, no significant differences were found at 5 and 10 min ($P > 0.05$) for each root sealer. Orange and eucalyptus oil solvents were as effective as chloroform at 2 min in dissolving all the root sealers.

Conclusions Xylol was the most effective solvent followed by the chloroform and the essential oils (eucalyptol and orange oil). Orange oil behaved in a similar way to eucalyptus oil.

Keywords: endodontic sealers, organic solvents, weight loss.

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Introduction

After endodontic therapy, the persistence of microbial infection in the root canal system or periradicular region is the major cause of failure (Nair *et al.* 1999, Siqueira 2001). Management of post-treatment pathosis includes root canal retreatment, that has good survival rates (Salehrabi & Rotstein 2010) and is less invasive in most cases (Karabucak & Setzer 2007).

The techniques used to remove root fillings include hand, mechanical and/or ultrasonic instruments (Ladley *et al.* 1991, Friedman *et al.* 1992, Scelza *et al.* 2008, Ring *et al.* 2009), heat (Ezzie *et al.* 2006) and laser irradiation (Anjo *et al.* 2004, Tachinami & Katsuumi 2010), either alone or in combination with solvents.

Organic solvents have been used to aid removal of gutta-percha and sealer (Martos *et al.* 2006, Magalhães *et al.* 2007). Laboratory studies have shown the effectiveness of various solvents against different types of endodontic sealers. For example, chloroform and

Correspondence: Josué Martos, Department of Semiology and Clinic, Faculty of Dentistry, Federal University of Pelotas, R. Gonçalves Chaves, 457, 96.015-560, RS, Brazil (Tel.: +55 53 32214162, fax: +55 53 32224439; e-mail: josue.sul@terra.com.br).

xylol have been shown to dissolve most root filling materials (Tamse *et al.* 1986, Wennberg & Orstavik 1989, Görduysus *et al.* 1997, Whitworth & Boursin 2000, Schäfer & Zandbiglari 2002, Magalhães *et al.* 2007), but it is reported to have carcinogenic potential and toxicity to tissues (Vajrabhaya *et al.* 2004).

Essential oils are able to dissolve most endodontic sealers and some, such as orange oil, eucalyptus oil and pine oil, have been reported to be safe and useful for this purpose (Hunter *et al.* 1991, Uemura *et al.* 1997, Hansen 1998). Ribeiro *et al.* (2007) when studying the biocompatibility of endodontic solvents reported that chloroform and eucalyptol were cytotoxic. Orange oil has been shown to be more biocompatible than eucalyptol, xylol, chloroform and halothane (Scelza *et al.* 2006). Orange oil acts on gutta-percha and sealer cements in the same way as xylol, without the deleterious effects (Oyama *et al.* 2002, Martos *et al.* 2006).

The behaviour of orange oil in comparison with eucalyptol, chloroform and xylol to dissolve zinc oxide–eugenol-based sealer is similar (Hansen 1998, Martos *et al.* 2006, Scelza *et al.* 2008, Ring *et al.* 2009). However, the ability to dissolve resin-based materials using orange oil or eucalyptol is thought to be poor (Hansen 1998, Schäfer & Zandbiglari 2002) although this view has been challenged with reports claiming it is as effective as chloroform (Bodrumlu *et al.* 2008, Ring *et al.* 2009) and xylol (Martos *et al.* 2006). With the increasing use of resin-based sealers is important to verify the action of solvents on these materials. Thus, the aim of this study was to analyse in a laboratory setting the solubility of different root canal sealers in organic solvents used in root canal retreatment.

Materials and methods

Calcium hydroxide-based/Sealer 26 (Dentsply Maillefer, Ballaigues, Switzerland), silicon polydimethylsiloxane-based/RoekoSeal (Coltène/Whaledent, Langenau, Germany), zinc oxide–eugenol-based/Endomethasone (Septodont, Saint-Maur-des-Fossés, France), resin-based/Epiphany (Pentron Clinical Technologies, LLC, Wallingford, CT, USA) and epoxy resin-based/EZ-Fill (Essential Dental Systems, South Hackensack, NJ, USA) (Table 1) sealers were used. Sealer cements were mixed in accordance with the manufacturers' instructions.

Freshly mixed materials were introduced into standardized stainless steel moulds with 8 mm diameter and 2 mm height, and a microscope slide was then pressed onto the upper surface to make the surface flat. Ten minutes after the mixture was prepared, the moulds were transferred to a chamber with 80% relative humidity and 37 ± 1 °C temperature for 72 h. Then, they were removed from the chamber,

Table 1. Compositions of the root canal sealers.

Sealer	Type of sealer	Manufacturer	Batch	Components
RoekoSeal	Silicone-based sealers	Coltène/Whaledent, Langenau, Germany	6001935/2012–07	Polydimethylsiloxane, silicone oil, paraffin, hexachloride platinum acid, zirconium dioxide
Sealer 26	Calcium hydroxide based	Dentsply/Maillefer, Petrópolis, RJ, Brazil	344830C/2013–05	Bismuth oxide, calcium hydroxide, hexamethylenetetramine, titanium dioxide. bisphenol epoxy resin
Epiphany	Resin-based cement	Pentron Clinical Technologies, LLC, Wallingford, CT	202396/2012–03	Bisphenol-A-glycidyldimethacrylate, polyethylene glycol dimethacrylate, ethoxylated bisphenol-A, dimethacrylate, urethane dimethacrylate, barium sulphate, silica, calcium oxide, bismuth, pigments
EZ-Fill	Epoxy resin based	EDS – Essential Dental Systems, South Hackensack, NJ	121809A/2011–12	Bisphenol-A epoxy resin, silver and bismuth oxide
Endomethasone	Zinc Oxide–Eugenol	Septodont, Saint-Maur-des-Fossés, Paris, France	47024AB/2012–10	Zinc oxide, dexamethasone, hydrocortisone acetate, diiodothymol, barium sulphate, trioxymethylene, magnesium estearate

and the excess material was then trimmed to the surface level of the mould with a scalpel and a brush. The samples were weighed in milligrams (up to four decimal places) on a precision scale (Sartorius ED124S, Sartorius AG, Göttingen, Germany) prior to immersion in the solvent to obtain the initial mass (m_1). The weights were recorded in duplicate.

Seventy-five samples of each endodontic sealer were prepared and divided into five groups of 15. The groups were further divided into three subgroups of five each according to immersion period (2, 5 and 10 min). The selected solvents were eucalyptus oil (SS White, Rio de Janeiro, RJ, Brazil), orange oil (Orangeform, Formula & Ação, São Paulo, SP, Brazil), xylol (Sigma-Aldrich Inc., St Louis, MO, USA), chloroform (Sigma-Aldrich Inc., St Louis, MO, USA) and distilled water (Milli-Q, Millipore Corp., Billerica, MA, USA).

Sealer specimens were immersed in 20 mL of solvent stored in an amber glass bottle with a screw cap (Corning Inc., New York, NY, USA) at room temperature. The immersion was such that both surfaces of each specimen were readily accessible to the solvent. Distilled water, obtained from a Milli-Q water system (Millipore Corp., Billerica, MA, USA), was used as a negative control. After the specified immersion period, the specimens were removed from the glass vial with the aid of tweezer with silicone tip, rinsed with 100 mL of double-distilled water and then blotted dry with absorbent paper. Samples were allowed to dry in an oven for 24 h at 37 ± 1 °C and then kept in a dehumidifier/desiccator. Thereafter, they were weighed (m_2), and the amount of lost sealer from each specimen was determined as the difference between the original weight of the sealer and its final weight.

The means and standard deviations of dissolution (weight loss) in grams were calculated at each time interval for each group of specimens (Table 2). The values were compared by factorial analysis of variance (ANOVA) using SPSS 18.0 software (SPSS Inc., Chicago, IL, USA), and the difference amongst the materials was calculated. Multiple comparison intervals were further performed to identify statistically homogeneous subsets ($P < 0.05$) using post hoc Student–Newman–Keuls, with the value of statistical significance set at 0.05.

Results

Dissolution means and standard deviations recorded for sealers immersed in different solvents are summarized in Table 2. In general, xylol had a significantly superior ability for dissolving root canal sealers in comparison

Table 2 Means (± standard deviations) of weight loss (mg) for each endodontic sealer at different solvents and time

	Orange oil			Eucalyptus oil			Xylol			Chloroform			Distilled water		
	2 min	5 min	10 min	2 min	5 min	10 min	2 min	5 min	10 min	2 min	5 min	10 min	2 min	5 min	10 min
RoekoSeal	$0.40^{A,a}$ (±0.10)	$0.80^{AB,a}$ (±0.10)	$0.80^{AB,a}$ (±0.30)	$0.50^{A,a}$ (±0.20)	$0.60^{AB,a}$ (±0.50)	$1.30^{ABC,a}$ (±0.40)	$2.60^{C,a}$ (±0.20)	$4.60^{D,a}$ (±0.50)	$5.50^{D,a}$ (±0.30)	$1.90^{ABC,a}$ (±0.10)	$1.80^{ABC,a}$ (±0.30)	$2.20^{BC,a}$ (±0.20)	$0.40^{A,a}$ (±0.10)	$0.50^{A,a}$ (±0.10)	$0.50^{A,a}$ (±0.10)
Sealer 26	$13.8^{A,b}$ (±0.40)	$14.9^{AB,b}$ (±0.50)	$16.4^{AB,b}$ (±0.90)	$17.4^{AB,b}$ (±0.50)	$17.9^{BC,b}$ (±0.20)	$18.2^{BC,b}$ (±0.90)	$22.0^{DE,b}$ (±0.30)	$24.6^{E,b}$ (±0.50)	$20.9^{CD,b}$ (±0.50)	$16.4^{AB,b}$ (±0.80)	$15.1^{AB,b}$ (±0.90)	$22.4^{DE,bc}$ (±0.10)	$1.40^{F,b}$ (±0.10)	$1.40^{F,b}$ (±0.10)	$1.70^{F,b}$ (±0.10)
Epiphany	$27.3^{A,c}$ (±0.60)	$27.4^{A,c}$ (±0.40)	$28.0^{A,c}$ (±1.70)	$28.1^{A,c}$ (±1.20)	$28.7^{AB,c}$ (±1.80)	$28.2^{A,c}$ (±0.60)	$33.8^{C,c}$ (±4.50)	$33.8^{C,c}$ (±0.40)	$33.5^{C,c}$ (±0.50)	$29.3^{AB,c}$ (±0.70)	$31.3^{BC,c}$ (±2.90)	$30.1^{AB,c}$ (±0.50)	$3.20^{D,c}$ (±0.10)	$3.20^{D,c}$ (±0.10)	$3.20^{D,c}$ (±0.10)
EZ-Fill	$32.0^{A,cd}$ (±0.20)	$33.9^{A,cd}$ (±0.60)	$29.7^{A,c}$ (±0.30)	$28.1^{A,c}$ (±0.60)	$27.7^{A,c}$ (±0.90)	$31.9^{A,c}$ (±0.20)	$31.6^{A,c}$ (±0.60)	$60.8^{B,ef}$ (±0.40)	$63.7^{B,f}$ (±0.10)	$32.2^{A,c}$ (±0.50)	$50.5^{B,d}$ (±0.20)	$60.0^{B,de}$ (±0.90)	$3.30^{C,c}$ (±0.20)	$3.60^{C,c}$ (±0.90)	$3.70^{C,c}$ (±0.20)
Endomethasone	$36.8^{A,d}$ (±1.0)	$42.3^{AB,e}$ (±1.0)	$46.5^{B,e}$ (±0.80)	$35.9^{A,c}$ (±1.0)	$45.4^{B,d}$ (±0.60)	$47.6^{B,d}$ (±0.50)	$49.4^{B,d}$ (±0.50)	$58.1^{C,e}$ (±2.0)	$60.6^{C,ef}$ (±2.0)	$49.4^{B,d}$ (±1.40)	$57.8^{C,de}$ (±1.40)	$63.6^{C,e}$ (±3.0)	$2.70^{D,c}$ (±0.10)	$2.80^{D,c}$ (±0.10)	$2.90^{D,c}$ (±0.10)

Means followed by the same superscript uppercase letter in rows indicate no statistically significant difference among the solvents for each endodontic sealer ($P < 0.05$). In columns, same superscript lower-case letter indicates no statistically significant difference among the endodontic sealer for each solvent ($P < 0.05$).

with the other solvents ($P < 0.05$). In the distilled water control group, minimum values of sealer dissolution were observed.

In the xylol solvent group, there was no significant difference for weight loss at 5 and 10 min ($P > 0.05$) for RoekoSeal, Epiphany, EZ-Fill and Endomethasone. Endomethasone and EZ-Fill had significantly more weight loss than the other three sealers at 10 min ($P < 0.05$).

In the chloroform solvent group, there was no significant difference for weight loss at 2 and 10 min ($P > 0.05$) for RoekoSeal, Sealer26 and Epiphany root canal sealers. Endomethasone and EZ-Fill had significantly more weight loss than the other sealers at 5 and 10 min in the chloroform group ($P < 0.05$).

In the eucalyptus oil solvent group, for each sealer, there was no significant difference for weight loss at 2, 5 and 10 min ($P > 0.05$) with exception of Endomethasone, which had significantly more weight loss than the other sealers at 5 and 10 min in this group ($P < 0.05$).

In the orange oil group, there was no significant difference for weight loss at 2, 5 and 10 min ($P > 0.05$) for RoekoSeal, Sealer26 and Epiphany root canal sealers. Endomethasone had significantly more weight loss at 5 and 10 min, respectively ($P < 0.05$). Endomethasone had significantly more weight loss than the other sealer at 10 min in this solvent group ($P < 0.05$). Epiphany and EZ-Fill had similar results for the three immersion times.

Discussion

The results of the present study indicate that the five root canal sealers were soluble to some degree in the four solvents. Xylol followed by chloroform was the more effective solvent on the different sealers tested followed by the essential oils (eucalyptol and orange oil).

The zinc oxide–eugenol-based sealer had the greatest solubility when compared to other cements as reported previously (Martos *et al.* 2006). Scelza *et al.* (2008) when comparing, by scanning electron microscopy, the efficacy of orange oil, eucalyptol and chloroform to dissolve zinc oxide–eugenol-based sealer (Endofill, Dentsply, RJ, Brazil) on the removal of filling materials from dentinal tubules during root canal retreatment reported that there was no significant difference amongst them. Nevertheless, Whitworth & Boursin (2000) observed that some zinc oxide–eugenol-based sealers are more soluble in chloroform than eucalyptol.

Sealer26, characterized as a resin-based sealer with calcium hydroxide in its composition, had low levels of dissolution with the solvents tested, corroborating previous studies (Martos *et al.* 2006). Calcium hydroxide-based sealers had a solubility between 5.3 and 7.3% of their initial weight when immersed in eucalyptol for 20 min (Schäfer & Zandbiglari 2002). Whitworth & Boursin (2000) reported a low solubility of calcium hydroxide cements in chloroform. In this study, xylol at 5 min and chloroform at 10 min were the most effective and were not significantly different; eucalyptol and orange oil at 10 min were similar to chloroform at 5 min. This small change in its final weight could be explained by the fact that the material contains calcium hydroxide that absorbs water, making a balance between disintegration and weight loss and water sorption and weight increase.

EZ-Fill suffered more degradation in xylol and chloroform at 10 min in comparison with other resin-based sealers. Interestingly, eucalyptol and orange oil showed similar results to xylol and chloroform at 2 min. One possible explanation for the different values obtained at 5 and 10 min may be due to the chemical composition of Epiphany, which contains more resinous elements than EZ-Fill (see Table 1).

Under the experimental conditions, RoekoSeal sealer had little change in eucalyptol and orange oil at the three time periods. However, a significantly higher value of dissolution was observed with the use of xylol for 5 and 10 min ($P < 0.05$). Possibly, its low solvency is explained because it is a silicone-based material. Schäfer & Zandbiglari (2002) reported values of solubility of RoekoSeal significantly lower in chloroform than in eucalyptol. Bodrumlu *et al.* (2008) assessed the solubility of Epiphany sealers, AH Plus and Ketac-Endo in the solvents chloroform and eucalyptol and reported that Epiphany had greater solubility in chloroform than in eucalyptol.

In several previous studies, chloroform was reported to be the solvent with the greatest capacity for dissolving most endodontic sealers (Whitworth & Boursin 2000, Schäfer & Zandbiglari 2002). Ring *et al.* (2009) found that orange oil and chloroform had similar results against AH Plus and RealSeal. In this sense, Bodrumlu *et al.* (2008) reported that eucalyptus oil or chloroform dissolved Epiphany and AH Plus to the same extent.

Conclusions

Xylol was the most effective solvent on the different sealers tested followed by the chloroform and the essential oils (eucalyptol and orange oil). Orange oil

behaved in a similar way to eucalyptus oil. Essential oils (eucalyptol and orange oil) at 5 and 10 min were similar to chloroform at 2 min.

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