

## RESEARCH ARTICLE

# Eucalyptol, Orange Oil, and Xilodent Solubility on Three Endodontic Sealants: An *in vitro* Study

<sup>1</sup>Yuri Rodriguez, <sup>2</sup>Frank Mayta-Tovalino

## ABSTRACT

**Aim:** To compare the *in vitro* solubility of eucalyptol, orange oil, and xilodent on three endodontic sealers based on their commercial brand and immersion time.

**Materials and methods:** A total of 240 samples of endodontic sealants were prepared, 80 from each group: I (EndoFill<sup>®</sup>), II (EndoSeal<sup>™</sup>), and III (Sealer 26<sup>®</sup>), and then they were divided into groups of 20 based on the type of solvent, eucalyptol, orange oil, xilodent, and sodium chloride (control group), which were immersed for periods of 5 and 10 minutes; the sample was calculated through the comparison of the means. Each specimen was weighted with an analytical bascule (Ohaus, USA) before and after each immersion.

**Results:** Comparisons were made on each group through the Student's t test. Eucalyptol revealed a higher dissolving effect in EndoSeal<sup>™</sup> followed by orange oil, and for xilodent at both immersion periods. It was found that there was only a statistically significant difference in the group eucalyptol-EndoSeal<sup>™</sup> in the period of immersion of 5 minutes with  $p = 0.033$ ; none of the three dissolvents had the capacity of dissolving the Sealer 26<sup>®</sup> during the different immersion periods.

**Conclusion:** The eucalyptol, orange oil, and xilodent can be used for deobturation of EndoFill<sup>®</sup> and EndoSeal<sup>™</sup> with variations on the specified times established by the manufacturer; however, the Sealer 26<sup>®</sup> was not soluble in the tested solvents.

**Keywords:** Dissolvent effect, Immersion time, Sealer cement.

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## INTRODUCTION

For the success of an endodontic treatment or retreatment, the establishment of a tridimensional filling of all the ramifications of the canal systems must be taken into

account as an essential requirement<sup>1</sup> so the persistence of a microbial infection of the canal systems or periapical area is prevented.<sup>2</sup> When endodontic therapy fails, the first alternative should be a nonsurgical approach, in contrast with other more radical approaches.<sup>3</sup> In the market, there is a variety of sealant materials for the filling of an endodontic treatment; the most used method for this process is based on the usage of semi-solid cones of gutta-percha as a base material; however, individually, they are unable to seal the root canal, and this is where the endodontic sealers complement the covering of dentin and fill the irregularities between the walls of a root canal, creating a properly sealed obturation.<sup>4</sup> Along history, different sealants have been developed based on different components, such as: zinc oxide, glass ionomer, calcium hydroxide, silicones, and composites,<sup>5,6</sup> and for the elimination of this obturation material (gutta-percha and sealant) hand instruments could be used, both mechanic and ultrasonic, as well as thermal techniques.<sup>7</sup> Additionally, different dissolvents have been used for the softening of the obturation materials.<sup>8,9</sup> The commonly used dissolvent, such as chloroform and xylol had the capacity of dissolving the majority of endodontic filling materials; their efficacy to soften filling materials has been shown in previous studies by Whitworth and Boursin,<sup>10</sup> Schäfer and Zandbiglari,<sup>11</sup> Magalhães et al,<sup>9</sup> Martos et al,<sup>12</sup> Mushtaq et al,<sup>13</sup> Fadhil and Al-Hashimi,<sup>1</sup> Yadav et al.<sup>14</sup> Nonetheless, they present a carcinogenic potential and toxicity for the tissues,<sup>15</sup> which is the reason why there is an interest to find alternative dissolvent that lacks adverse effects for the health of the patients; therefore, some organic alternatives have appeared, such as orange oil and eucalyptol, with capacity of softening endodontic materials, a safe alternative to chloroform and its derivatives.<sup>1,8,16</sup> Presenting different compositions, the endodontic sealants also present different physical characteristics that may influence the clinical efficacy of the dissolvent. Hence, the aim of the present study was to compare the *in vitro* dissolvent effect of eucalyptol, orange oil, and xilodent on different endodontic sealers based on different brands and the immersion time.

## MATERIALS AND METHODS

Each of the tested cements was prepared following the manufacturer's instructions: base of zinc oxide-eugenol/

<sup>1</sup>Dental Surgeon, <sup>2</sup>Professor and Academic Coordinator

<sup>1</sup>Department of Stomatology School, Universidad Privada San Juan Bautista Lima, Peru

<sup>2</sup>Department of Masters in Public Health, Universidad Privada San Juan Bautista, Lima, Peru

**Corresponding Author:** Frank Mayta-Tovalino, Professor and Academic Coordinator, Department of Masters in Public Health Universidad Privada San Juan Bautista, Lima, Peru, Phone: +511013464822, e-mail: frank.mayta@upsjb.edu.pe

**Table 1:** Composition of the endodontic sealers

Endodontic sealer	Type of sealer	Manufacturer	Batch #	Components
EndoFill®	Zinc oxide–eugenol	Dentsply/ Maillefer, Argentina	054549G/2017-12	Zinc oxide, hydrogenated resin, subcarbonate of bismuth, barium sulfate and sodium borate, eugenol, sweet almonds oil
Sealer 26®	Calcium hydroxide and bismuth oxide by epoxy resin	Dentsply/ Maillefer, Brazil	137086H/2018-10	Bismuth trioxide, calcium hydroxide, hexamethylene tetramine, titanium dioxide, epoxy bisphenol
EndoSeal™	Zinc oxide–eugenol with dexamethasone	Prevest DenPro, India	1181605/2019-03	Zinc oxide, subcarbonate of bismuth, barium sulfate, dexamethasone, thymol iodide, hydrocortisone acetate, magnesium stearate, eugenol, epoxy resin

EndoFill (Dentsply Maillefer, Argentina), base of zinc oxide–eugenol with dexamethasone/EndoSeal™ (Prevest DenPro, India), base of calcium hydroxide/Sealer 26® (Dentsply Maillefer, Brazil), and their compositions are shown in Table 1. Each sealer cement was mixed according to the manufacturer's instructions.

The sealers were carefully poured in the sample molds with a 1 mL syringe preventing the formation of bubbles, all samples with their respective molds were transferred to a chamber (Mettler, Germany) with 80% of relative humidity and a temperature 37°C, and were stored for 24 hours.

After removing the samples from the chamber, the excessive material was removed with a scalpel, and thereafter, each sample was weighed in grams including up to four decimals (3 times each sample) using an analytical scale (Ohaus, USA), obtaining the initial weight (p1). The samples of the groups I, II, and III were divided into four groups of 20 samples each, based on the dissolvent to use: Xilodent (Proquident, Colombia), orange oil (Maquira, Brazil), eucalyptol (Maquira, Brazil), and sodium chloride (Braun, Peru) as a control group; then, each group was subdivided into 2 groups of 10 for the immersion times of 5 and 10 minutes.

After being weighted and divided, every sample of sealer was completely immersed in 40 mL of dissolvent previously stored in Petri dishes; after a period of immersion specified by group of 5 or 10 minutes, the samples were removed using cotton tweezers; subsequently, each sample was rinsed with 100 mL of distilled water and dried with an absorbent paper.

The samples were dried for 24 hours at a temperature of 37°C in the chamber. To finish, each sample was weighed (3 times) obtaining a final weight (p2), and the amount of sealer lost on every sample obtained from the difference between the initial and the final weight was determined.

For the elaboration of the univariate analysis, we proceeded to obtain the measures of means and standard deviations of the variable "dissolvent effect" of xilodent, orange oil, and eucalyptol based on the established groups (Sealer 26®, EndoFill® and EndoSeal™) and the

covariables of the study (commercial brand and immersion time). Additionally, the normality of the sample was determined through Shapiro–Wilk test.

For the bivariate analysis, Student's t test was used to compare the dissolvent effect on the different study groups based on the commercial brand and the immersion time; the results are shown in Table 2.

All the analyses were performed with the Stata® statistical package, version 12.0, with a significance level of  $p < 0.05$ .

## RESULTS

Once the *in vitro* effect of dissolvent of the orange oil, xilodent, eucalyptol, and sodium chloride on the groups was formed by EndoFill®, EndoSeal™, and Sealer 26®, it was evident that a higher dilution was obtained in the 10-minute immersion groups in comparison with the 5-minute groups. When the Shapiro–Wilk normality test was analyzed, it was shown that every group presented a normal distribution with  $p > 0.05$ . In the statistical inference to the effect of the dissolvent through Student's t-test on the groups formed by the sealer cements, it was shown that eucalyptol had a higher dissolving effect than EndoSeal™, followed by orange oil, and then by xilodent in both immersion periods; it was evident that in the eucalyptol-EndoSeal group measuring the difference between the initial weight and the final weight after 5 minutes of immersion,  $p = 0.03$ ; in the control group of sodium chloride, minimal dissolution values were observed. The eucalyptol was also capable of dissolving the EndoFill®, but this time followed by the xilodent and then by the orange oil.

None of the three dissolvents had the capacity to dissolve Sealer 26® during the different immersion periods; the results are shown in Table 2.

## DISCUSSION

The specific standards to measure the dissolution capacity of endodontic sealers are unavailable; therefore, the present study was conducted aiming to assess the *in vitro*

**Table 2:** In vitro comparison of the dissolution effect of Eucalyptol, orange oil, and xilodent on different endodontic sealers by time and brand

Sealer	Min	Orange oil			Eucalyptol			Xilodent					
		Initial weight	Final weight	p*	p**	Initial weight	Final weight	p*	p**	Initial weight	Final weight	p*	p**
Endofill®	5	0.2460 ± 0.05	0.2434 ± 0.05	0.569	0.457	0.3011 ± 0.04	0.2919 ± 0.04	0.712	0.336	0.2718 ± 0.04	0.2694 ± 0.04	0.553	0.456
	10	0.2772 ± 0.04	0.2739 ± 0.04	0.690	0.434	0.2836 ± 0.04	0.2721 ± 0.04	0.677	0.298	0.2682 ± 0.05	0.2610 ± 0.04	0.930	0.375
Endoseal™	5	0.3614 ± 0.03	0.3045 ± 0.04	0.995	0.209	0.2821 ± 0.04	0.2464 ± 0.04	0.503	0.033	0.2728 ± 0.05	0.2718 ± 0.05	0.632	0.484
	10	0.3427 ± 0.04	0.2888 ± 0.04	0.642	0.155	0.3188 ± 0.05	0.2800 ± 0.05	0.549	0.070	0.2989 ± 0.07	0.2948 ± 0.07	0.631	0.452
Sealer 26®	5	0.3220 ± 0.04	0.3218 ± 0.04	0.150	0.497	0.3455 ± 0.06	0.3452 ± 0.06	0.15	0.495	0.3275 ± 0.07	0.3274 ± 0.07	0.270	0.498
	10	0.3156 ± 0.06	0.3153 ± 0.06	0.897	0.496	0.3933 ± 0.07	0.3928 ± 0.07	0.552	0.494	0.3399 ± 0.07	0.3396 ± 0.07	0.260	0.497

All the measures were performed in grams; statistical significance level p < 0.05; p\* Shapiro Wilk; p\*\* Student's t test

effect of eucalyptol dissolvent, orange oil, xilodent and sodium chloride, on the more used commercially available sealers in the dental market: EndoFill®, EndoSeal™, and Sealer 26®.

The criterion and methodology involved in the present study to measure the dissolution of the endodontic sealers on the dissolvent were through the calculation of weight differences during the pre- and postimmersion process. These data were collected with an analytical scale with the capacity of finding the weight in grams including up to four decimals.<sup>1,12-14</sup>

Some previous studies have used different immersion times, due to the fact that previous investigations have reported that a mean time of 1.5 to 10.8 minutes is required for the elimination of the obturation materials by the instrumentation with or without the aid of a dissolvent.<sup>10,14,17</sup> The present study was performed with immersion times of 5 and 10 minutes; in that way, measures were more precise using a sample of each sealer cement for every immersion period.

After each immersion period, every sample was rinsed in distilled water to eliminate unwanted debris.<sup>1,18</sup> It was needed to dry the samples for 24 hours, because during the pilot test, an increase in the postimmersion weight in some specimens was shown. The data obtained from the present study showed that orange oil presented higher efficacy after 10 minutes than after 5 minutes, showing a higher dissolving effect in the EndoSeal™, because it showed a higher weight loss after 5 and 10 minutes of immersion, in comparison with their counterparts EndoFill®, in a second position, and Sealer 26®, with a lower weight loss after 5 and 10 minutes of immersion, concluding that the aforementioned dissolvent did not present the dissolution capacity required for endodontic sealers based on calcium hydroxide and bismuth oxide by epoxy resin, results similar to the publication of Schäfer and Zandbiglari.<sup>11</sup> Some authors, such as Yadav et al<sup>14</sup> and Ring et al<sup>19</sup> mentioned a similar efficacy to dissolve the endodontic sealers with base of zinc oxide and eugenol, and also the difficulties they encountered to dissolve the resin-based endodontic sealers.<sup>14,19</sup> These results differ from the findings of Mushtaq et al,<sup>13</sup> who used Apexit® Plus (calcium hydroxide), AH Plus® (epoxy resin), and Endoflas® (zinc oxide and eugenol) to assess the dissolvent effect of the xilen, refined orange oil, tetrachloroethylene, and distilled water (control group); the data obtained showed that xilen was the dissolvent with a better performance in dissolving the endodontic sealer followed by refined orange oil and tetrachloroethylene.

Regarding the dissolving effect of the eucalyptol on tested the endodontic sealers, the data have shown that there was a higher dissolving effect after 10 minutes than

during 5 minutes of immersion, and EndoSeal™ was the cement that showed the greatest dissolution in comparison with EndoFill®, which had a moderate dissolution, and the cement Sealer 26® showed the lower dissolution on both immersion periods. There was a statistically significant difference in the immersion time of 5 minutes of EndoSeal™, when compared with the immersion time of the same cement for 10 minutes, likewise in the same immersion times of 5 and 10 minutes of EndoFill® and Sealer 26®. These data differ from the findings presented by Fadhil and Al-Hashimi,<sup>1</sup> who assessed the dissolving effect of xilen, eugenate desobturator, eucalyptol, ethylenediaminetetraacetic acid (EDTA), and distilled water (as control group) on EndoFill®, Apexit® Plus, and EndoSequence, concluding that xilen is the dissolvent with higher capacity to dissolve EndoFill® and the rest of the endodontic sealers above eucalyptol, even though the latter has also shown dissolution capacity for EndoFill®. Martos et al<sup>12</sup> used eucalyptol and orange oil to measure the dissolution of Sealer 26® on dissolution times of 2, 5, and 10 minutes, showing that this dissolvent did not show significant differences on any immersion time mentioned, corresponding with the findings of the present research.

The data after the immersion of the samples of xilodent revealed that there was a higher dissolving effect in the EndoFill®, above EndoSeal™ in the second position and Sealer 26® in the last position in both immersion periods of 5 and 10 minutes. The dissolution found during the immersion time of 10 minutes was higher than the immersion for 5 minutes in EndoFill® and EndoSeal™; in Sealer 26®, there was no substantial dissolution when comparing the initial with the final weight in both immersion periods. Previous investigations have determined that the xylol or xilen is the gold standard of endodontic dissolvent due to the destabilization of covalent bonds that link carbon atoms; this matches the conclusions obtained by Fadhil and Al-Hashimi,<sup>1</sup> who evaluated the solubility of xilen, eugenate, eucalyptol, EDTA, and distilled water (control group) in four different kinds of endodontic sealers EndoFill®, Apexit® Plus, AH Plus®, and bioceramic sealer EndoSequence, and their results revealed that the four dissolvents can be used to eliminate the EndoFill® and Apexit® Plus. Martos et al<sup>8</sup> aimed to assess the solubility of three types of endodontic sealers: Sealer 26® (calcium hydroxide), RoekoSeal (silicium-polydimethylsiloxane), EndoFill®, and Intrafill (zinc oxide–eugenol) on eucalyptol, xylol, orange oil and distilled water (control group), obtaining as results that xylol and orange oil showed better dissolving effects. The researchers assessed the endodontic sealers based on zinc oxide to assess the dissolving effect of xilen, showing that xilen was capable of dissolving this sealer cement.

The limitations of using this methodology of measuring the dissolving effect of xilodent, orange oil, and eucalyptol in sealer cements EndoFill®, EndoSeal™, and Sealer 26® are the following: it was not possible to compare clinically relevant parameters, such as temperature of the dissolvent in the oral cavity, the diverse anatomy of the canal systems, the dissolution of the agents on the existing biological fluids, and the advantage or disadvantage that could be presented with the aid of manual techniques, rotatory instrumentation, ultrasound, etc. Fadhil and Al-Hashimi<sup>1</sup> and Keleş<sup>20</sup> agreed with this idea of clinically relevant parameters that were not possible to assess in the present study.

## CONCLUSION

- The present study found statistically significant difference when assessing the dissolving effect of eucalyptol on the EndoSeal™ group, measured by the difference between the initial and the final weight after 5 minutes of immersion.
- The other groups described in the present report did not show a significant dissolving effect on the different immersion times.

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