

ORIGINAL RESEARCH

Solubility Evaluation of Different Root Canal Sealing Materials

¹Emre Bayram, ²Dilek Dalat, ³Melike Bayram

ABSTRACT

Aim: To evaluate the solubility of five different root canal sealers (AH Plus Jet, RealSeal SE, MTA Fillapex, Tubli-Seal, and Acroseal) in chloroform, eucalyptol and Endosolv-E solvents.

Materials and methods: Ninety root canal sealer samples were prepared and then divided into three groups for immersion in a solvent for 2, 5 or 10 minutes. The mean values of the root canal sealers' dissolution in the solvents were obtained by the difference between the preimmersion original weight and the post-immersion weight on a digital analytical scale. Data were statistically analyzed by a Kruskal-Wallis test with a Bonferroni correction.

Results: Chloroform was a more effective solvent than eucalyptol or Endosolv E for all root canal sealers, except for RealSeal SE, at all time points ($p < 0.003$). RealSeal SE was the least soluble sealer in all solvents at all time points.

Conclusion: Chloroform demonstrated a superior ability over other solvents in dissolving canal sealing materials, and eucalyptol was found to be the least effective solvent in this study.

Clinical significance: This study can help to the clinicians about which solvent should be preferred for solving the filling materials in retreatment cases.

Keywords: Endodontic sealers, Retreatment, Solubility, Solvents.

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INTRODUCTION

A combination of gutta-percha and root canal sealers are commonly used in root canal filling materials. Gutta-percha used without a root canal sealer is not adequate to completely fill the root canal system.¹ A paste-type

sealer is essential as both a binding agent and to fill irregularities; sealers cement the gutta-percha to the dentin and can be used to fill accessory canals, anastomoses and multiple foramina.²

Teeth may need to be retreated for a variety of reasons. In cases of inadequate cleaning or filling of the root canal system, procedural errors, or reinfection of the primary sealed root canal caused by coronal or apical leakage, retreatment is indicated.³⁻⁵ A large portion of the gutta-percha and sealers should be removed in order to reach the bacteria and tissue residues that are the causes of endodontic failure.

To date, many published reports have illustrated that it is difficult to completely eliminate filling materials from the walls of the major canals during retreatment; it is presumably even more difficult to clean microscopic residues.⁶⁻⁸

Different methods are used to remove root canal filling materials. These methods include the heat method,⁹ hand tools, mechanical and ultrasonic instruments used in mechanical methods,¹⁰ laser irradiation,¹¹ and chemicals. Chemical methods, either alone or in combination, are preferred for the removal of canal filling materials.¹² Removal techniques are dependent upon canal size and anatomy, condensation degree, and quantity of gutta-percha.^{8,13} Sealer used in conjunction with gutta-percha may remain inaccessible to mechanical removal when located in anatomical ramifications.^{14,15} In such cases, solvents are essential for a thorough cleaning of the filling material from the root canal system, debris removal, and to provide effective disinfection.¹⁵

The removal of gutta-percha by mechanical means can lead to unwanted outcomes such as root perforation and changing the original shape of the root canal. Therefore, the use of solvent greatly facilitates the removal of filling material.⁴ An ideal solvent for endodontic retreatment balances the requirements of an acceptable level of clinical safety, level of toxicity and tissue destruction, and chemical capacity for dissolution.¹⁶

The purpose of this *in vitro* study was to evaluate the solubility of five different root canal sealers (AH Plus, RealSeal SE, MTA Fillapex, Tubli-Seal and Acroseal) in different solvents (chloroform, eucalyptol and Endosolv E).

^{1,3}Department of Endodontics, Gaziosmanpasa University Tokat, Turkey

²Department of Endodontics, Ankara University, Ankara Turkey

Corresponding Author: Emre Bayram, Assistant Professor Department of Endodontics, Gaziosmanpasa University, Tokat Turkey, Phone: 03562300036, e-mail: bayremre@yahoo.com

MATERIALS AND METHODS

Epoxy resin-based AH Plus Jet (Dentsply De Trey, Konstanz, Germany), calcium hydroxide-based Acroseal (Septodont, Saint-Maur-des-Fosses Cedex, France), zinc oxide and eugenol-based Tubli-Seal (Kerr, USA), MTA-based MTA Fillapex (Angelus Industria de Produtos Odontologicos S/A, Londrina—PR. Brasil), and resin-based RealSeal SE (SybronEndo, Glendora, USA) were tested in this study.

Stainless steel ring molds with an internal diameter of 8 mm and a height of 2 mm were used to prepare 90 samples of root canal filling material. All sample molds were cleaned with acetone in an ultrasonic bath for 15 minutes and weighed in triplicate prior to use (AND GR— 202, Tokyo, Japan). All weight measurements throughout the study were in grams recorded to five decimal places. Molds were supported by a larger glass plate and covered with a cellophane sheet. Sealer cements were mixed in accordance with the manufacturer's instructions, and then freshly mixed materials were carefully flowed to excess into the sample molds, avoiding air entrapment. Another glass plate, also covered with cellophane film, was positioned on the mold and pressed manually in such a way that the plates were in contact with the entire mold in a uniform manner. The assembly was placed in an incubator (37°C, 95% relative humidity) and left to set for 24 hours. Excess material was then trimmed level with the surface of the mold with a sharp scalpel. Before the immersion of the samples, all sealers were weighed in their molds three times and the average value was recorded (m_1).

Ninety samples of each endodontic sealer were prepared and divided into three groups of 30. The groups were further divided into three subgroups of 10, according to the immersion period (2, 5 or 10 minutes). The selected solvents were eucalyptol (Dentsply, Trappes, France), chloroform (Aklar Kimya, Ankara, Turkey), and

Endosolv E (Septodont, Saint-Maur-des-Fosses Cedex, France).

At room temperature, sealer samples were immersed in 20 ml of solvent. Both surfaces of the samples were accessible to the solvent. We removed the sealer samples from the solvents after the specified immersion period by using a pair of tweezers, touching only the metal mold. Then, the samples were washed with 100 ml of double-distilled water and then blotted dry with absorbent paper. Samples were allowed to dry in an oven for 24 hours at $37 \pm 1^\circ\text{C}$ and then kept in a dehumidifier/desiccator. Thereafter, they were weighed (m_2), and the amount of sealer removed from the specimen was determined as the difference between the original weight of the sealer and its final weight. The weight loss of each sample, expressed as a percentage of the original mass, was considered the solubility of the tested material.

The differences in dissolution of root canal sealers for different durations and in different solvents were tested and calculated using Kruskal-Wallis tests with Bonferroni corrections ($p < 0.003$). Kruskal-Wallis tests with Bonferroni corrections were also further performed to compare the amount of dissolution of the root canal sealers, with the level of statistical significance set at $p < 0.006$.

RESULTS

Dissolution means and standard deviations are reported as percentages for the sealers immersed in different solvents and presented in (Table 1).

Chloroform was a more effective solvent than eucalyptol or Endosolv E for all root canal sealers, with the exception of RealSeal SE, at all time points ($p < 0.003$ (Graph 1).

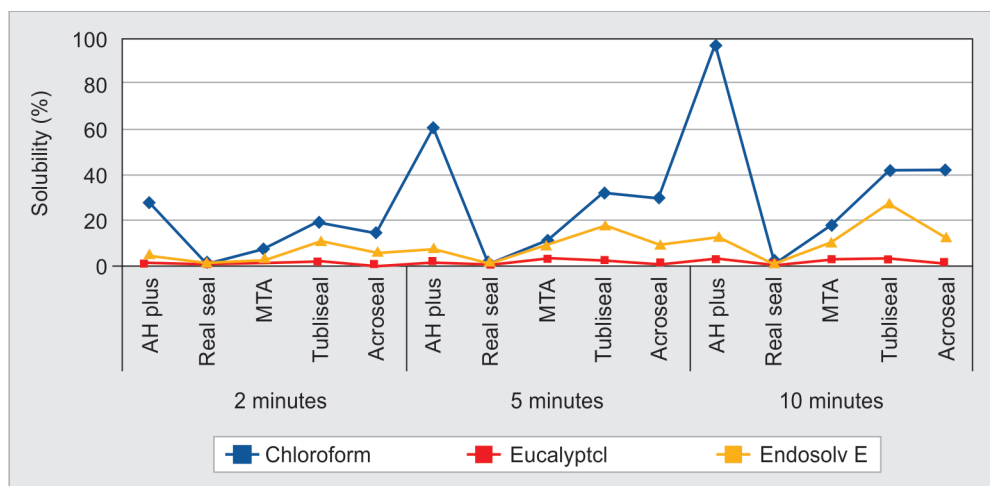
DISCUSSION

In a study by Bodrumlu¹⁷ the solubilities of AH Plus, Epiphany, and Ketac-Endo sealers were tested in

Table 1: The mean percentage (\pm standard deviation) of weight loss for each endodontic sealer in different solvents over time

	Chloroform			Eucalyptol			Endosolv E		
	2 min.	5 min.	10 min.	2 min.	5 min.	10 min.	2 min.	5 min.	10 min.
AH Plus	27.53 ^{A,a} (± 3.46)	60.34 ^{B,a} (± 7.00)	97.28 ^{C,a} (± 2.65)	0.73 ^{C,bc} (± 0.44)	1.58 ^{A,ab} (± 0.59)	2.79 ^{B,a} (± 0.57)	4.99 ^{B,bc} (± 1.91)	7.92 ^{C,c} (± 1.91)	13.22 ^{A,abc} (± 2.27)
RealSeal	0.33 ^{A,d} (± 0.20)	0.62 ^{A,d} (± 0.31)	0.72 ^{A,e} (± 0.31)	0.34 ^{A,cd} (± 0.09)	0.35 ^{A,c} (± 0.20)	0.49 ^{A,c} (± 0.32)	0.34 ^{A,d} (± 0.27)	0.33 ^{A,d} (± 0.26)	0.56 ^{A,d} (± 0.45)
MTA	7.53 ^{A,cd} (± 1.56)	11.03 ^{B,cd} (± 3.44)	17.38 ^{C,de} (± 5.73)	1.36 ^{C,ab} (± 0.35)	3.11 ^{A,a} (± 1.14)	3.15 ^{A,a} (± 1.46)	2.58 ^{D,cd} (± 0.63)	9.04 ^{B,bc} (± 7.94)	10.44 ^{B,c} (± 7.85)
Tubli seal	19.23 ^{A,ab} (± 3.64)	31.54 ^{B,ab} (± 4.12)	41.65 ^{C,c} (± 3.77)	1.70 ^{D,a} (± 0.82)	2.07 ^{D,a} (± 0.60)	2.87 ^{A,a} (± 0.56)	11.06 ^{C,a} (± 2.27)	18.03 ^{A,a} (± 1.70)	27.66 ^{B,a} (± 1.92)
Acroseal	14.36 ^{A,bc} (± 0.66)	30.10 ^{B,bc} (± 1.85)	41.69 ^{C,bc} (± 2.20)	0.12 ^{B,d} (± 0.05)	0.56 ^{D,bc} (± 0.23)	0.86 ^{D,bc} (± 0.45)	5.68 ^{C,abc} (± 0.83)	9.05 ^{A,abc} (± 1.27)	11.85 ^{B,bc} (± 1.06)

The means that are followed by the same superscript uppercase letter in the rows indicate no statistically significant difference among the solvents for each endodontic sealer ($p < 0.003$). In the columns, the same superscript lower-case letter indicates no statistically significant difference among the endodontic sealers for each solvent ($p < 0.006$)



Graph 1: The mean percentage of weight loss for each endodontic sealer in different solvents over time

eucalyptus oil, chloroform, and distilled water as a control. It was found that there was no statistically significant difference between the root canal sealers at 2 or 5 minutes, and the epiphany root canal sealer showed statistically greater solubility than the AH Plus sealer at 10 minutes in the solvents.

Epiphany and Epiphany SE are dual-curable resin composites containing a redox catalyst.¹⁸ RealSeal SE and Epiphany SE have the same structure as fourth-generation self-etching methacrylate resin-based root canal sealers. The manufacturer of epiphany states that 40 seconds of light is required to cure the coronal region of the sealer, whereas the entire filling will cure chemically in approximately 15 to 30 minutes.

In a study by Resende¹⁹ even though epiphany and epiphany SE were mixed and manipulated in a darkroom during the experiment, a thin, superficial, non-cured layer was always observed after the required setting time after the specimens were exposed to a curing light source. According to Franco,²⁰ oxygen inhibited vinyl polymerization in composite resins, and 40 to 60% of the carbon bonds remained unsaturated. The lack of uniform photoactivation throughout the sample contributes to incomplete polymerization, leaving residual monomers in the sealer in the deepest recesses of the sample.²¹ Compared to the results of Bodrumlu,¹⁷ our results indicate that RealSeal SE was the least soluble root canal sealer in the solvents tested. This difference may be resulted from the insufficient polymerization of the epiphany sealer. Mathias-Junior,²² observed that the solubilities of light-activated epiphany and uncured epiphany sealers were significantly different in distilled water. Photoactivation may cause an interlocking of the polymeric chains, creating a more rigid and closed structure and thus, hindering the mobility of Ca^{2+} ions inside the sealer matrix.

In our study, RealSeal SE showed less solubility than other root canal sealers. This result may be because

RealSeal SE has a more rigid structure after light activation. However, study by Martos,¹² supported our results regarding RealSeal SE. They found that there was no significant difference in weight at 2, 5 or 10 minutes for RealSeal SE in chloroform or eucalyptus oil.

Chloroform was a more effective solvent than eucalyptol for the MTA Fillapex root canal sealer. However, MTA Fillapex dissolved more than other root canal sealers in eucalyptol. There are few published reports on retreatment efficiency following the use of this material, or on the actions of other solvents. In spite of this, there are no reports of MTA Fillapex solubility in different organic solvents.^{23,24} In our findings, MTA Fillapex had the greatest solubility in chloroform at 2, 5 and 10 minutes. In addition, eucalyptol was found to be the least effective solvent for this root canal sealer.

In several previous studies, AH Plus demonstrated greater solubility than calcium hydroxide-based sealers or a zinc oxide and eugenol-based sealers in chloroform solvent.²⁵ In the present study, almost all of the AH Plus had dissolved at 10 minutes, and the solubility of AH Plus was higher than that of Acroseal or Tubli-Seal root canal sealer in chloroform. Our results were similar to those of Whitworth²⁵ and Schafer.²⁶ Schafer²⁶ observed that AH Plus was less soluble than $\text{Ca}(\text{OH})_2$ and eugenol-based sealers in eucalyptus oil that contained 85% eucalyptol.

Tanomaru-Filho²⁷ showed that the solvent action of eucalyptol was effective on eugenol-based sealers, but no action was found on the resin-based sealers AH Plus, Epiphany, or EndoREZ, or on the silicone-based sealer Roekoseal. These findings support the results of our study. According to our results, Tubli-Seal was significantly more soluble than Acroseal, but the difference was not significant between AH Plus and Tubli-Seal at 5 or 10 minutes in the eucalyptol solvent group.

Tetrachloroethylene is the main compound in Endosolv E, which has been used to dissolve zinc oxide and

eugenol sealers.²⁸ This study showed that the solubility of Tubli-Seal was higher than that of other root canal sealers in Endosolv E at all time points.

A reduction in solubility and the disintegration of zinc oxide and eugenol-based (ZnOE) cements when natural resin was added to the composition.²⁹ However, Martos³⁰ observed that most ZnOE-based filling cements have vegetable resin in their compositions; they are composed of approximately 90% resinous acids and are soluble in solvents commonly used for endodontic retreatment.

In previous studies, researchers have reported that calcium hydroxide-based sealers showed a low level of solubility in solvents. The solubility of Acroseal was less than that of the Tubli-Seal root canal sealer in eucalyptol. This may result from the relative insolubility of its ingredients; it contains bisphenol A diglycidyl ether and methenamine, which are epoxy compounds found in the structures of AH 26 and Sealer 26.^{25,30}

CONCLUSION

Chloroform displayed a superior ability in dissolving canal sealing materials over the other solvents tested, and the least solvent capacity was obtained with eucalyptol. AH Plus was the most soluble sealer in chloroform solvent and almost all of the AH Plus had dissolved in chloroform at 10 minutes. RealSeal SE was the least soluble sealer in all solvents and at all time points. The solubility of all canal filling sealers, with the exception of RealSeal SE, significantly increased with increasing time.

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