

# An Evaluation of the Solubility of Four Endodontic Sealers in Different Solvents (An *In Vitro* Study)

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## ABSTRACT

**Background:** Complete removal of filling material from the root canal is an essential requirement for endodontic retreatment. The purpose of the present study is to evaluate and compare the dissolving capabilities of various solvents (Xylene, Eugenate Desobturator, Eucalyptol, EDTA and Distilled water (as a control)) on four different types of sealer (Endofill, Apexit Plus, AH Plus and EndoSequence bioceramic sealer).

**Materials and method:** Eighty samples of each sealer were prepared according to the manufacturers' instructions and then divided into ten groups (of 8 samples) for immersion in the respective solvents for 2 and 5 min immersion periods. Each sealer specimen was weighed to obtain its initial mass. The specimens were immersed in the tested solvents for 2 and 5 min, followed by rinsing with double distilled water and blotted dry with an absorbent paper, then they were reweighed to determine its final mass. The mean of weight loss was determined for each material in each solvent during the specified immersion period, and the values were subjected to statistical analysis.

**Results:** Clear differences were shown in the solubility profile of these root canal sealers in the tested solvents. The result of the present study shows that Xylene had the greatest capacity for dissolving Endofill, Apexit Plus and AH Plus. Eugenate Desobturator, Eucalyptol and EDTA showed a highly significant dissolving capability on these sealers with variations between these subgroups; EndoSequence BC sealer is insoluble in these tested solvents. Regarding the immersion time, higher values of solubility were obtained at 5 min than that at 2 min immersion time.

**Conclusion:** The results showed that Xylene, Eugenate Desobturator, Eucalyptol and EDTA can be used for the removal of Endofill, Apexit Plus and AH Plus during endodontic retreatment with variations between these subgroups; D.W (control group) showed the least capacity for dissolving these sealers. EndoSequence BC sealer is insoluble in the tested solvents.

**Keywords:** Endofill, Apexit Plus, AH Plus, EndoSequence BC sealer, Xylene, Eugenate Desobturator, Eucalyptol, EDTA, Endodontic retreatment, Solvents. (J Bagh Coll Dentistry 2015; 27(4):15-20).

## INTRODUCTION

Teeth with pulpal and periradicular disease are usually treated with root canal treatment. Although, the success rate of root canal treatment is up to 86 to 93%, failure in endodontic treatment may be expected. The main causes of root canal failure are improper cleaning and filling of the root canal system, procedural errors, or the lack of an efficient hermetic sealing, which enables the survival of bacteria inside dentinal tubules, apical ramifications, accessory and secondary canals<sup>(1)</sup>.

Retreating previously filled root canal requires that antimicrobial irrigants and medications gain access to all anatomical ramifications of the canal system which may be harboring microorganisms and organic matter. It is desirable that all materials employed are amenable to complete removal during retreatment. Failure to remove all debris from the canal may result in the survival of bacterial infection, which may result in root canal failure<sup>(2,3)</sup>.

The most commonly used obturation material in endodontic treatment is gutta-percha in conjunction with a variety of sealers. To allow endodontic retreatment when indicated, the root filling materials should be retreatable/retrievable<sup>(4)</sup>.

Various removal methods are available for endodontic retreatment, including mechanical instrumentation alone or in combination with the solvents or heat. While methods for removing the gutta-percha have been well researched, far less interest has been focused on the removal of sealer from root canal walls, and from root ramification where they remain inaccessible to mechanical techniques of removal. In such cases, solvents are essential for the thorough cleaning of filling material/debris to allow effective disinfection of the root canal system<sup>(5)</sup>.

The 'wicking technique' is essential in removing residual gutta-percha and sealer and should always be the final step during gutta-percha removal. Wicking technique involve flushing the root canal with a solvent up to the level of pulp chamber followed by drying it with paper points. Paper points aid in removal of residual materials by drawing dissolved materials into and then out of the shaped canal<sup>(1,6)</sup>.

Chloroform and Xylene have the ability to dissolve most root filling materials. As a result of concerns about the carcinogenicity of chloroform, researchers and clinicians have an interest in finding alternative solvents. Some solvents act as safe alternative to chloroform such as orange oil and eucalyptol with ability to dissolve root filling materials<sup>(7)</sup>.

Several commercially available endodontic sealers present with distinct physicochemical

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characteristics, which may determine and influence the clinical efficiency of the solvents. Therefore, in the present study we evaluated the dissolving capabilities of various solvents on different types of sealer.

## MATERIALS AND METHODS

For this in vitro study, standardized metal ring (8 mm in diameter and 2 mm in height) were used to prepare the sealer specimens. Eighty samples were prepared from each sealer material.

- Group A: 80 samples of Endofill sealer (zinc oxide eugenol-based sealer).
- Group B: 80 samples of Apexit Plus sealer (Calcium hydroxide-based sealer).
- Group C: 80 samples of AH plus sealer (Epoxy resin based sealer).
- Group D: 80 samples of EndoSequence BC sealer (calcium phosphate silicate-based sealer).

Each group divided into ten subgroups (of 8 samples) for immersion in the solvents for 2 min and 5 min. Sealers prepared according to the manufacturers' instructions. After loading the molds, they transferred to a humidifier with 80% relative humidity and  $37\pm 1^\circ\text{C}$  temperature for 72 hours (EndoSequence BC sealer remained in

humidity for 10 days to be completely set). Then they removed from the humidifier and excess material trimmed with a scalpel blade to the surface level of the mold<sup>(8)</sup>.

Every sample removed from the mold in such a way that all surfaces of each sample were freely accessible to the liquid. The samples weighed in grams (up to four decimal places) by using a digital analytical scale (Sartorius Analytical, Germany) prior to the immersion in the solvent to get the initial weight. The sealer samples immersed in the respective solvents (20 ml) for 2 and 5 minute immersion periods.

Each sample used for just one immersion period to enhance the accuracy of the measurements. After the specific immersion period, the samples removed from the container, rinsed with 100 ml of double-distilled water in order to neutralize the solvent action. After that, the specimens blotted dry with an absorbent paper to remove the loose debris of decomposition<sup>(8)</sup>. The sealer samples allowed to dry in an oven for 24 hours at  $37\pm 1^\circ\text{C}$ . Thereafter, they weighed to obtain the final weight.

Lost from each sample calculated by the difference between the final weight and the original weight of the sample.

## RESULTS

The descriptive statistics (mean and standard deviation) and t-test results of weight loss (g) for each sealer presented in the tables (1,2,3,4).

**Table 1: Descriptive Statistics and t-test Results of weight Loss (g) for Group A (Endofill Sealer).**

Solvents	Groups	Descriptive statistics				Difference			
		Mean	$\pm$ S.D.	Mean	$\pm$ S.D.	Mean difference	t-test	d.f.	p-value
Xylene	Wa-W <sub>2</sub>	0.274	0.003	0.259	0.003	0.015	37.543	7	0.000
	Wb-W <sub>5</sub>	0.274	0.002	0.255	0.005	0.019	15.208	7	0.000
	Diff.	0.0146	0.0012	0.0190	0.0035	-0.004	-3.336	14	0.005
Eugenate Desobturator	Wa-W <sub>2</sub>	0.275	0.003	0.263	0.003	0.011	18.940	7	0.000
	Wb-W <sub>5</sub>	0.274	0.001	0.255	0.003	0.020	18.291	7	0.000
	Diff.	0.0112	0.0016	0.0196	0.0030	-0.008	-6.863	14	0.000
EDTA	Wa-W <sub>2</sub>	0.272	0.003	0.266	0.003	0.007	25.382	7	0.000
	Wb-W <sub>5</sub>	0.274	0.002	0.264	0.003	0.010	26.101	7	0.000
	Diff.	0.0066	0.0007	0.0100	0.0011	-0.003	-7.280	14	0.000
Eucalyptol	Wa-W <sub>2</sub>	0.274	0.002	0.270	0.002	0.004	12.503	7	0.000
	Wb-W <sub>5</sub>	0.275	0.001	0.266	0.001	0.009	34.942	7	0.000
	Diff.	0.0042	0.0010	0.0092	0.0007	-0.005	-11.520	14	0.000
Distilled Water	Wa-W <sub>2</sub>	0.275	0.001	0.275	0.001	0.0001	1.930	7	0.095
	Wb-W <sub>5</sub>	0.275	0.001	0.275	0.001	0.0001	7.514	7	0.000
	Diff.	0.0001	0.0001	0.0001	0.0001	0.000	-2.016	14	0.063

**Table 2: Descriptive Statistics and t-test Results of Weight Loss (g) for Group B (Apexit Plus)**

Solvents	Groups	Descriptive statistics				Difference			
		Mean	±S.D.	Mean	±S.D.	Mean difference	t-test	d.f.	p-value
Xylene	Wa-W <sub>2</sub>	0.194	0.0005	0.168	0.0041	0.026	20.363	7	0.000
	Wb-W <sub>5</sub>	0.195	0.0011	0.145	0.0034	0.050	35.956	7	0.000
	Diff.	0.0261	0.0036	0.0503	0.0040	-0.024	-12.769	14	0.000
Eugenate Desobturator	Wa-W <sub>2</sub>	0.194	0.0003	0.178	0.0014	0.016	30.202	7	0.000
	Wb-W <sub>5</sub>	0.195	0.0009	0.150	0.0046	0.045	23.672	7	0.000
	Diff.	0.0165	0.0015	0.0447	0.0053	-0.028	-14.358	14	0.000
EDTA	Wa-W <sub>2</sub>	0.195	0.0009	0.193	0.0008	0.001	9.010	7	0.000
	Wb-W <sub>5</sub>	0.195	0.0005	0.193	0.0005	0.002	11.559	7	0.000
	Diff.	0.0011	0.0003	0.0020	0.0005	-0.001	-4.395	14	0.001
Eucalyptol	Wa-W <sub>2</sub>	0.195	0.0008	0.173	0.0031	0.021	16.059	7	0.000
	Wb-W <sub>5</sub>	0.196	0.0013	0.168	0.0035	0.028	21.262	7	0.000
	Diff.	0.0214	0.0038	0.0281	0.0037	-0.007	-3.546	14	0.003
Distilled Water	Wa-W <sub>2</sub>	0.195	0.0005	0.195	0.0005	0.0001	2.376	7	0.049
	Wb-W <sub>5</sub>	0.195	0.0007	0.195	0.0007	0.0002	6.110	7	0.000
	Diff.	0.0001	0.0001	0.0002	0.0001	0.000	-3.274	14	0.006

**Table 3: Descriptive Statistics and t-test Results of Weight Loss (g) for Group C (AH Plus).**

Solvents	Groups	Descriptive statistics				Difference			
		Mean	±S.D.	Mean	±S.D.	Mean difference	t-test	d.f.	p-value
Xylene	Wa-W <sub>2</sub>	0.334	0.001	0.310	0.008	0.024	8.416	7	0.000
	Wb-W <sub>5</sub>	0.334	0.001	0.300	0.002	0.034	42.442	7	0.000
	Diff.	0.0256	0.0072	0.0339	0.0023	-0.008	-3.081	14	0.008
Eugenate Desobturator	Wa-W <sub>2</sub>	0.334	0.001	0.331	0.001	0.003	12.261	7	0.000
	Wb-W <sub>5</sub>	0.334	0.001	0.330	0.001	0.004	27.710	7	0.000
	Diff.	0.0030	0.0006	0.0044	0.0004	-0.001	-5.103	14	0.000
EDTA	Wa-W <sub>2</sub>	0.334	0.001	0.334	0.001	0.000	14.279	7	0.000
	Wb-W <sub>5</sub>	0.334	0.001	0.334	0.001	0.001	10.095	7	0.000
	Diff.	0.0005	0.0001	0.0005	0.0002	0.000	-1.203	14	0.249
Eucalyptol	Wa-W <sub>2</sub>	0.336	0.002	0.331	0.003	0.005	9.673	7	0.000
	Wb-W <sub>5</sub>	0.335	0.001	0.328	0.002	0.007	16.406	7	0.000
	Diff.	0.0053	0.0015	0.0069	0.0012	-0.002	-2.310	14	0.037
Distilled Water	Wa-W <sub>2</sub>	0.335	0.000	0.335	0.000	-	-	-	-
	Wb-W <sub>5</sub>	0.335	0.001	0.335	0.001	-	-	-	-
	Diff.	0	0	0	0	-	-	-	-

**Table 4: Descriptive Statistics and t-test Results of Weight Changes (g) for Group D (EndoSequence BC)**

Solvents	Groups	Descriptive statistics				Difference			
		Mean	±S.D.	Mean	±S.D.	Mean difference	t-test	d.f.	p-value
Xylene	Wa-W <sub>2</sub>	0.255	0.001	0.258	0.003	-0.002	-2.181	7	0.066
	Wb-W <sub>5</sub>	0.255	0.001	0.256	0.001	-0.001	-7.417	7	0.000
	Diff.	-0.0025	0.0032	-0.0007	0.0003	-0.002	-1.536	14	0.147
Eugenate Desobturator	Wa-W <sub>2</sub>	0.256	0.001	0.261	0.001	-0.006	-25.731	7	0.000
	Wb-W <sub>5</sub>	0.256	0.001	0.264	0.001	-0.009	-34.350	7	0.000
	Diff.	-0.0057	0.0006	-0.0089	0.0007	0.003	9.540	14	0.000
EDTA	Wa-W <sub>2</sub>	0.256	0.001	0.257	0.001	-0.001	-9.565	7	0.000
	Wb-W <sub>5</sub>	0.256	0.001	0.259	0.001	-0.003	-5.289	7	0.001
	Diff.	-0.0013	0.0004	-0.0030	0.0016	0.002	2.951	14	0.011
Eucalyptol	Wa-W <sub>2</sub>	0.255	0.001	0.256	0.001	-0.001	-6.509	7	0.000
	Wb-W <sub>5</sub>	0.256	0.001	0.257	0.001	-0.001	-6.347	7	0.000
	Diff.	-0.0010	0.0004	-0.0011	0.0005	0.0002	0.639	14	0.533
Distilled Water	Wa-W <sub>2</sub>	0.255	0.001	0.257	0.001	-0.002	-11.745	7	0.000
	Wb-W <sub>5</sub>	0.255	0.001	0.257	0.001	-0.002	-28.987	7	0.000
	Diff.	-0.0015	0.0004	-0.0015	0.0001	0.0000	0.090	14	0.930

Wa: Original weight (g) for the 2-min. group.  
Wb: Original weight (g) for the 5-min. group.

W<sub>2</sub>: Sample weight (g) after 2 min immersion time.  
W<sub>5</sub>: Sample weight (g) after 5 min immersion time.

## DISCUSSION

Endodontic retreatment requires the complete debridement of the remnants of filling materials; for removing these fillings and sealer out of the aberration and fins of root canal systems, literature has proposed “wicking action” to be a final step in removing the root filling, this can be provided by solvents. Hence, it will be useful to use solvents with hand or rotary files for removing root canal debris<sup>(1,9)</sup>. The present study conducted to comparatively evaluate the dissolving capabilities of different solvents (Xylene, Eugenate Desobturator, Eucalyptol and EDTA) on four root canal sealers (Endofill, Apexit Plus, AH Plus and EndoSequence BC sealer).

Xylene is chlorinated hydrocarbon commonly considered as a common solvent of organic substances, possibly because of destabilization of the covalent bonds linking the carbon atoms<sup>(10)</sup>. It may also dissolve or soften the root canal sealers and could potentially facilitate their removal by mechanical means<sup>(1)</sup>. In 1992 Pécora et.al. presented orange oil, it is an essential oil used as a dissolving oil of zinc oxide eugenol-based cement. Orange oil represents an excellent alternative solvent when compared to potentially toxic solvents<sup>(8)</sup>.

Eucalyptol, the major component of eucalyptus oil, it is used in pharmaceuticals for fragrance, flavoring and to increase appetite (refreshing flavor). Eucalyptol clinically acceptable solvent, and are not considered potentially cancerogenic or cytotoxic<sup>(7)</sup>. A study in 2009 stated that EDTA used for removing the smear layer can also dissolve some sealers that are possible to remain in root canals. Therefore, the above four mentioned solvents used in the present study. Distilled water used as a control group in the study<sup>(11)</sup>.

Endodontic sealer divided according to their chemical composition into: zinc oxide-eugenol based, calcium hydroxide based, glass ionomer based, resin-based, bioceramic based, MTA-based and silicon based sealer<sup>(12)</sup>. In the present study, four types of root canal sealers from different chemical groups are selected. Endofill is a zinc oxide eugenol-based sealer. Traditionally, ZOE-based sealers have been the most commonly used sealants. They act as the gold standard against which other types of sealers are compared, since they reasonably have most of Grossman's requirements for root canal sealers. Apexit Plus is calcium hydroxide based-sealers.

No studies were found for evaluating and compare the dissolution of Apexit Plus sealer in (Xylene, Eugenate Desobturator, Eucalyptol and

EDTA). AH plus is epoxy resin based sealers, it is mechanically harder and more difficult in removal than zinc oxide eugenol-based sealers. A study in 2002 have stated that resin-based sealers can attach more strongly to both gutta-percha and dentin as compared to zinc oxide eugenol and calcium hydroxides based sealers<sup>(13)</sup>. A study in 2007 have mentioned that resin-based sealers have more consistent and deeper penetration into dentinal tubules than other types of sealers both in vitro and in vivo<sup>(14)</sup>.

In paint industries, solvents can often used for softening resin coating materials on paints to permit their easy removal. These solvents that used for paints removal can be considered in endodontic retreatment to remove the strongly adhering resin-based sealer from root canal walls<sup>(1)</sup>. Whitworth and Boursin evaluated the solubility of AH Plus, Apexit and Tubli-Seal sealers in halothane and chloroform, they concluded that AH Plus sealer was significantly more soluble than other tested sealers in both halothane and chloroform<sup>(2)</sup>.

In the present study, the evaluation and comparison of the solubility of AH Plus in Xylene, Eugenate Desobturator, Eucalyptol, EDTA and distilled water were performed. EndoSequence BC sealer (calcium phosphate silicate-based sealer) a revolutionary premixed and injectable root canal sealer utilizing new bioceramic nanotechnology. A study in (2011) evaluated the efficacy of ProTaper universal retreatment instruments, hand files, heat and chloroform on the removal of BC sealer when used in combination with gutta-percha as compared with AH Plus sealer, it concluded that conventional retreatment methods are not able to completely remove BC sealer<sup>(15)</sup>. In the present study, we evaluated the effect of Xylene, Eugenate Desobturator, Eucalyptol and EDTA on EndoSequence BC sealer, because no other studies examined if these solvents are effective in removal of this sealer during root canal retreatment.

There is no international standard or tests to study the dissolution of root canal filling materials in solvents. The ISO 6876:2001 standard explains the procedure to evaluate the solubility of set sealer in water. According to the instructions, ring molds with an internal diameter of 20 mm and 1.5mm in height should be used<sup>(16)</sup>. Similar methods have been previously described<sup>(3,1,17,18,10)</sup> using different sizes of ring molds (5 x 2; 4 x 2; 8 x 2; 6.4 x 1.6mm). In the present study, standardized metal ring 8mm in internal diameter and 2mm in height used to prepare the sealer specimens.

Few clinical data are presented on the time clinicians typically leaves the root canal flooded with solvents during root canal retreatment. Laboratory-based revealed that the time required for removing material is approximately 2-10 minutes. Researchers in previous studies used the same immersion periods<sup>(11)</sup>. In the present study, 2 and 5 min immersion times are used.

In order to enhance the accuracy of the measurements, one sample used for just one immersion period, thus undesired weight loss of the specimens because of the repeated drying and immersion is excluded. Each sample immersed in new solvent to ensure the purity of the solvents.

After the immersion period, all sealer samples rinsed in double-distilled water in order to remove loose materials of decomposition<sup>(19)</sup>. Drying process during 24 hours was suitable to the methodology because in previous pilot study an increase in weight after the immersion detected in some samples<sup>(20)</sup>. The weight loss in each sealer sample calculated to determine their dissolution. The criteria to evaluate the amount of the lost material were according to a study of Martos et.al.<sup>(18)</sup>.

This method provides a simple, cost-effective and reproducible method of solubility evaluation<sup>(2)</sup>. It should be kept in mind that this method allows only the comparison between different materials<sup>(16)</sup>. In using this method, we could not consider several clinically relevant parameters such as temperature of solvents, canal system anatomy and dilution of solvent by biological fluids or irrigants because of in vitro conditions<sup>(11)</sup>.

The results from the present study showed that the endodontic sealers used in the study, except EndoSequence BC sealer, were soluble in the tested solvents, and there were differences among the groups. This result is in accordance with several previous studies<sup>(3, 11, 18,21, 22)</sup> who reported the ability of some solvents to dissolve root canal sealer during endodontic retreatment.

#### **The Dissolving Efficacy of Different Solvents on Group A (Endofill Sealer):**

The data of the present study provide evidence that Xylene is more effective for Endofill cement than other solvents; since it showed more dissolution value after 2min immersion time, followed by Eugenate Desobturator, EDTA and Eucalyptol in descending order, there is a highly significant difference between Xylene and other tested solvents ( $P < 0.01$ ); while after 5 min immersion time, Eugenate Desobturator showed the best dissolving capability followed by Xylene,

no significant difference between these two solvents ( $P > 0.05$ ).

Higher values of solubility obtained at 5 min than that at 2 min immersion time ( $P < 0.01$ ). D.W showed the least dissolving capability at both 2 and 5 min immersion times with a highly significant difference between D.W. and other tested solvents ( $P < 0.01$ ). These results are in agreement with<sup>(23)</sup> who found that Xylene and Orange oil had a similar effects, and there is significant solubilization of Endofill and Intrafill (zinc oxide eugenol-based sealers) for 2, 5 or 10 min immersion times.

#### **The Dissolving Efficacy of Different Solvents on Group B (Apexit Plus Sealer):**

Regarding the solubility of Apexit Plus in tested solvents, the data of the present study showed that there is a significant solubilization of Apexit Plus in all tested solution. Xylene is more effective solvents for Apexit Plus than other tested solvents, because it exhibited the best dissolving capability at both 2 and 5 min immersion times ( $P < 0.01$ ).

Eugenate Desobturator, Eucalyptol and EDTA had a highly significant dissolving capability at both 2 and 5 min ( $P < 0.01$ ) with variations among these subgroups. D.W showed the least dissolving capability, there is a high significant difference between the D.W and other tested solvents ( $P < 0.01$ ). Higher values of solubility obtained after 5 min than that after 2 min.

#### **The Dissolving Efficacy of Different Solvents on Group C (AH Plus Sealer):**

According to the results of the present study, there is a significant weight loss in all tested solvents except the control group at both 2 and 5min immersion times. Xylene is a far more effective solvent for AH Plus cement than other solvents; since it exhibited the best dissolving capability at both 2 and 5min immersion time. Sheno et, al. (2014) mentioned that the setting of epoxy resin-based sealers includes polymerization and cross linking of their monomers, producing 3D lattice. This set polymer is not affected by water or saline, maybe due to the existence of HEMA in its composition. Hydrophobic organic solvents such as Xylene may have the capability to penetrate the 3D lattice leading to swelling of the lattice and reducing the strength and hardness of the material<sup>(1)</sup>.

Eugenate Desobturator, Eucalyptol and EDTA had a highly significant dissolving capability at both 2 and 5 min ( $P < 0.01$ ) with variations between these subgroups. Higher values of solubility were obtained at 5 min.

### The Dissolving Efficacy of Different Solvents on Group D (EndoSequence BC Sealer):

The data after immersion of EndoSequence BC samples in the tested solvents showed that there is an increase in the weight of EndoSequence BC samples in all tested solvents with variations between these subgroups, possibly due to liquid sorption by its components. The mechanism related to liquid sorption and distribution in the matrix is still not totally elucidated.

With most materials, there are two competitive processes that take place, one is dissolution and the other is fluid uptake<sup>(19)</sup>. The results of the present study showed that the ability of EndoSequence BC sealer to absorb fluid is much greater than its rate of dissolution. From that it is obvious that EndoSequence BC sealer is insoluble in these tested solvents.

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