

Evaluation of softening ability of Xylene & Endosolv-R on three different epoxy resin based sealers within 1 to 2 minutes - an *in vitro* study

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Objectives: This study evaluated the efficacy of Endosolv-R and Xylene in softening epoxy resin based sealer after 1 to 2 min exposure. **Materials and Methods:** Sixty Teflon molds (6 mm x 1.5 mm in inner diameter and depth) were equally divided into 3 groups of 20 each. AH 26 (Dentsply/De Trey), AH Plus (Dentsply/De Trey), Adseal (Meta-Biomed) were manipulated and placed in the molds allotted to each group and allowed to set at 37°C in 100% humidity for 2 wk. Each group was further divided into 2 subgroups according to the solvents used, i.e. Xylene (Lobachemie) and Endosolv-R (Septodont). Specimens in each subgroup were exposed to respective solvents for 1 and 2 min and the corresponding Vicker's microhardness (HV) was assessed. Data was analysed by Mauchly's test and two-way analysis of variance (ANOVA) with repeated measures, and one-way ANOVA. **Results:** Initial hardness was significantly different among the three sealers with AH Plus having the greatest and Adseal having the least. After 2 min, Xylene softened AH Plus and Adseal sealer to 11% and 25% of their initial microhardness, respectively ($p < 0.001$), whereas AH 26 was least affected, maintaining 89.4% of its initial microhardness. After 2 min, Endosolv-R softened AH 26, AH Plus and Adseal to 12.7, 5.6 and 8.1% of their initial microhardness, respectively ($p < 0.001$). **Conclusions:** Endosolv-R was a significantly more effective short term softener for all the tested sealers after 2 min whereas Xylene was an effective short term softener against AH plus and Adseal but less effective against AH 26. (*Restor Dent Endod* 2014;39(1):17-23)

Key words: Adseal; AH 26; AH plus; Endosolv-R; Micro-hardness; Xylene

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Introduction

Teeth with pulpal and periradicular involvement are commonly treated with root canal treatment. Although success rate of endodontic treatment is up to 86 to 93%, failure in endodontic treatment can be expected.¹ The main causes of endodontic failure are insufficient cleaning, inadequate obturation, untreated or missed out root canals, lack of efficient hermetic sealing and survival of bacteria.² These make nonsurgical endodontic retreatment necessary. For effective results, retreatment requires thorough debridement of former root canal filling materials including sealers.³ Debridement especially in resin based sealers that strongly adhere to the root canal dentin is more difficult.^{4,5} The root canal filling material in bulk can be easily removed with hand and rotary instrument leaving small amount of residue attached to the root canal dentin. Recently, Duncan and Chong have suggested the use of solvents to remove this root

canal residue.⁵ The aim of this study was to evaluate the softening ability of two solvents Xylene & Endosolv-R on three Epoxy resin based endodontic sealers that will facilitate their effective mechanical removal.

Materials and Methods

Three epoxy resin based root canal sealers, AH 26 (Group I, Dentsply/De Trey, Konstanz, Germany), AH Plus (Group II, Dentsply/De Trey) and Adseal (Group III, Meta-Biomed, Cheongwon, Korea) were tested. Composition of each sealer is described in Table 1. A well of 6 × 1.5 mm in diameter and depth, respectively, was prepared in each of sixty Teflon disks of 12 × 2 mm in diameter and height, respectively. These sixty molds were randomly and equally divided into three groups, each containing 20 Teflon molds ($n = 20$). Each sealer was then mixed according to the manufacturer's instructions and placed into the well of the mold. The sealer specimens were then allowed to set for 2

weeks at 37°C and 100% humidity. The design of the sealer specimen was such that only one surface of the specimen was exposed to solvent. Twenty set sealer specimens in each group were further randomly and equally divided into two subgroups ($n = 10$) based on solvents to which they were exposed to Xylene (Subgroup A, Lobachemie, Mumbai, India) and Endosolv-R (Subgroup B, Septodont, Cedex, France).

Initial Vicker's microhardness (HV) of each fully set sealer specimens were calculated using a Mitutoyo microhardness testing machine (Instrument No. 810-117E, Mitutoyo, New Delhi, India) with a Vicker's microhardness indenter. The indenter was applied at three predetermined points on the specimen surface with the load of 10 grams for 10 seconds. The indentations in the sample surface were measured under 100 times magnification with the microscope attached to the same machine. The mean of three was taken for each sample.

Each sealer specimen was then immersed in a petridish

Table 1. Composition of three resin-based root canal sealers

Sealer	Manufacturer	Composition
AH 26 silver free	Dentsply-Detrey, Konstanz, Germany	AH 26 powder: Bismuth oxide, Methenamine AH 26 resin: Bisphenol epoxy resin
AH plus	Dentsply-Detrey, Konstanz, Germany	Epoxide paste Diepoxide Calcium tungstate Zirconium oxide Aerosil Pigment Amine paste 1-adamantane amine N,N'-dibenzyl-5-oxa-nonandiamine-1,9 TCD-Diamine Calcium tungstate Zirconium oxide Aerosil Silicone oil
Adseal	Meta-Biomed, Cheongwon, Korea	Base Epoxy Oligomer resin Ethylene glycol salicylate Calcium phosphate, Bismuth subcarbonate Zirconium oxide Catalyst Poly aminobenzoate Triethanolamine Calcium phosphate Bismuth subcarbonate Zirconium oxide Calcium oxide

containing the corresponding solvent (i.e. Xylene and Endosolv-R) for 1 minute. Each specimen was then retrieved from the solvent and air dried. At this point of time, the microhardness of each sealer specimen was reassessed by similar procedure as mentioned above. Each sealer specimen was then again exposed to corresponding solvents for additional 1 minute. (i.e. totally 2 minutes.) and similar procedure to evaluate microhardness was followed as mentioned above. In this way ten sealer specimens from each group were tested for reduction in Vicker's microhardness (HV) after 1 and 2 minutes of exposure to solvent.

Vicker's microhardness (HV) for each sealer at initial stage, after 1 and 2 minutes exposure to solvents was summarized (Tables 2 and 3) in terms of means and standard deviations. Reduction in the microhardness of each sealer at one and two minutes was expressed as percentage with reference to the respective initial hardness. To determine the effect of solvents and sealer types on the microhardness according to time, two-way analysis of variance (ANOVA) with repeated measures was performed. The assumption of sphericity was assessed using Mauchly's test. Additionally, to determine the significance of difference in the mean hardness across groups at each time point, one-way ANOVA was used followed by Tuckey's *post-hoc* pairwise comparison. The analysis was performed using SPSS 16.0 (SPSS INC., Chicago, IL, USA) software.

Results

The descriptive statistics in terms of mean and standard deviation were obtained for each sealer type according to time, for each solvent Xylene and Endosolv-R (Tables 2 and 3). It was evident that the mean Vicker's hardness reduced significantly with time for all sealer and solvent combinations. However, their extent of reduction was different. To assess the statistical relevance of change in the hardness due to sealer type and solvent exposure, two-way ANOVA with repeated measures was performed independently for two solvents.

Solvent-Xylene (Subgroup A)

Mauchly's sphericity test for each of the three effects in the model i.e. two main effects (group and time) and the interaction effect (group x time) revealed that the assumption of sphericity was met by group ($p = 0.32$) and hence no correction of F-ratios was required. However, time and interaction violated the assumption ($p < 0.05$). The estimate of sphericity (ϵ) for these effects were 0.543 and 0.385 respectively, and hence Greenhouse-Geisser correction was referred for degrees of freedom of F-statistics. The results of ANOVA revealed that there was a highly significant main effect of groups ($F_{(2,18)} = 591.48$, $p < 0.0001$) when exposed to Xylene. The main effect of

Table 2. Mean and standard deviation of Vicker's microhardness (HV; $n = 10$) of root canal sealers after exposure to Xylene

Time	Group I (AH 26)	Group II (AH Plus)	Group III (Adseal)
Initial hardness	147.71 ± 7.84 ^{ab}	157.31 ± 5.84 ^{aA}	122.54 ± 8.28 ^{cC}
Hardness after 1 min	135.21 ± 8.0 ^{bA} (8.5%)	21.85 ± 1.53 ^{bc} (86.1%)	42.85 ± 5.64 ^{bb} (65.1%)
Hardness after 2 min	132.05 ± 8.23 ^{cA} (10.6%)	17.32 ± 0.54 ^{cc} (89.1%)	30.52 ± 4.99 ^{cb} (75.1%)

For each sealer type (column), superscripted small letters indicate statistically significant difference in mean microhardness upon exposure to Xylene ($p < 0.05$) with time. Superscripted capital letters indicate statistically significant difference in mean hardness across groups. Values in the brackets indicate percentage reduction in the microhardness with reference to initial hardness.

Table 3. Mean and standard deviation of Vicker's microhardness (HV; $n = 10$) of root canal sealers after exposure to Endosolv-R

Time	Group I (AH 26)	Group II (AH Plus)	Group III (Adseal)
Initial hardness	139.61 ± 7.67 ^{ab}	163.92 ± 6.99 ^{aA}	121.91 ± 5.65 ^{cC}
Hardness after 1 min	52.36 ± 3.84 ^{bb} (62.5%)	63.29 ± 5.85 ^{bA} (61.4%)	37.54 ± 4.80 ^{bc} (69.2%)
Hardness after 2 min	17.69 ± 1.67 ^{cA} (87.3%)	9.23 ± 0.44 ^{cb} (94.4%)	9.88 ± 1.20 ^{cb} (91.9%)

For each sealer type (column), superscripted small letters indicate statistically significant difference in mean microhardness upon exposure to Endosolv-R ($p < 0.05$) with time. Superscripted capital letters indicate statistically significant difference in mean hardness across groups. Values in the brackets indicate percentage reduction in the microhardness with reference to initial hardness.

time was also highly significant ($F_{(1,08,9.77)} = 2,760.26$, $p < 0.0001$), indicating that the marginal mean hardness (HV) was different at three time points. The interaction effect was significant ($F_{(1.54,13.86)} = 1,115.52$, $p < 0.0001$) indicating that the hardness (HV) attained at different time points depends on the type of sealer material when exposed to Xylene (Table 2). Exposure time of Xylene had a noticeable effect on AH Plus followed by Adseal (Figure 1).

Solvent-Endosolv-R (Subgroup B)

Mauchly's sphericity test revealed that the two main effects, i.e. group and time as well as the interaction effect (group \times time) met the assumption of sphericity with p values 0.729, 0.389 and 0.174 ($p > 0.05$), respectively. Hence, there was no need of any correction of F-ratios. The ANOVA revealed that the main effect of groups was highly significant ($F_{(2,18)} = 114.44$, $p < 0.0001$). The main effect of time was also highly significant ($F_{(2,18)} = 11,208$, $p < 0.0001$) indicating that the marginal mean of hardness (HV) was different at three time points. The interaction effect (group \times time) was also significant ($F_{(4,36)} = 51.22$, $p < 0.0001$) suggesting the dependency of hardness (HV) of sealer type exposed to Endosolv-R and time (Table 3, Figure 2). The

mean Vicker's microhardness (HV) at different time points for AH 26 was higher than that of Adseal. However, AH Plus contributed mainly to the interaction effect. Mean hardness (HV) for this sealer when exposed to Endosolv-R was higher than that for other two sealers. However, after two minutes exposure, the mean hardness (HV) for AH Plus dropped remarkably with a mean of 9.23 ± 0.44 (94.4% reduction) and was very close to that of Adseal (9.88 ± 1.20). In short, the effect of Endosolv-R exposure to AH Plus was noticeable after two minutes.

The above analyses revealed that AH Plus sealer exposed to either of the solvents had the maximum reduction in the mean hardness (HV) as compared to other two sealers, and in particular, the effect is pronounced for Endosolv-R exposure to AH Plus. Additionally, one-way ANOVA in subgroup A suggested that mean initial hardness (HV) differed significantly across three groups. Similar was the observation after 1 and 2 minutes. Further, also in subgroup B, the mean initial hardness (HV) differed significantly in three groups, and the finding was consistent after 1 minute. However, after 2 minutes, the mean hardness (HV) of group I differed significantly than groups II and III, while the means of groups II and III showed no significant difference.

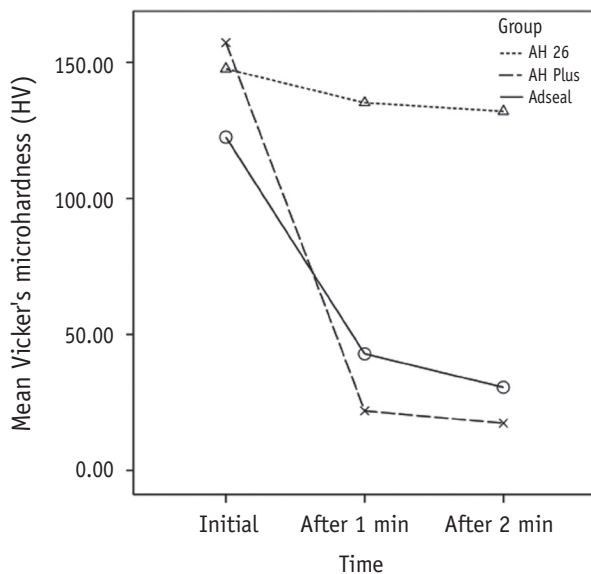


Figure 1. Mean Vicker's microhardness (HV) of three sealers exposed to Xylene at three time points.

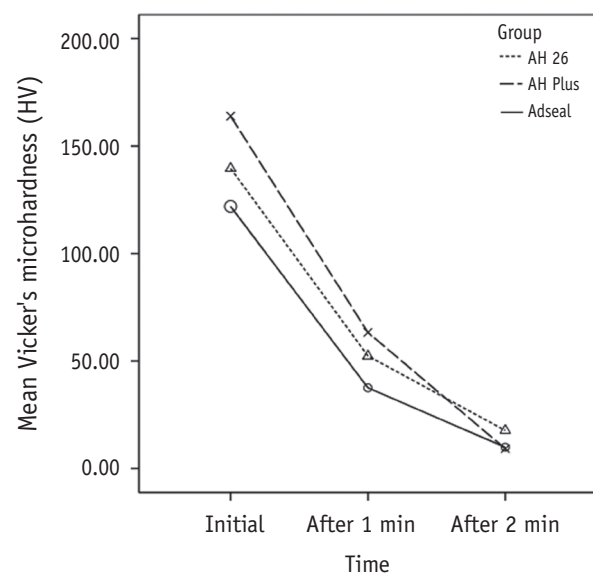


Figure 2. Mean Vicker's microhardness (HV) of three sealers exposed to Endosolv-R at three time points.

Discussion

For endodontic retreatment to be successful, it is necessary to completely remove all previous obturation material.^{3,6} An ideal root canal sealer should be easily removed if retreatment is necessary to allow access for antimicrobial agent and medicament to all root canal ramifications.⁷ Shin *et al.* advocated the use of Gates Glidden and Profile systems for retrieval of resin based root canal sealer.⁸ Recently, Duncan and Chong suggested many methods for removal of root filling materials including use of the hand files, rotary files, ultrasound, heated pluggers and solvents.⁵ Hand and rotary instruments are commonly being used for effective removal of root canal fillings.^{2,3} The bulk of these filling materials can be removed within 2 to 3 minutes, but still remnants of gutta percha and sealer remains commonly attached to root canal dentin.^{6,9} With better sealing and bonding, resin based root canal sealers are of interest to many clinicians.^{4,10,13} The complete debridement of the remnants of resin based sealers that strongly attaches to the dentin is prolonged tedious task. So to remove these fillings and sealer out of the fins and aberration of root canal systems, literature has suggested 'wicking action' is necessary which can be provided by solvents.^{5,11,12} Therefore, it will be helpful to use solvents along with hand and rotary files to remove root canal debris.

Three epoxy resin based sealers, AH plus, AH 26, and Adseal were used because they are mechanically harder and more difficult to remove than zinc oxide eugenol based ones.^{5,10,13} Lee *et al.* have mentioned that resin based sealers attach more strongly to both dentine and gutta percha as compared to zinc oxide eugenol and calcium hydroxides based ones.¹⁰ Mamootil *et al.* have stated that resin-based sealers have deeper and more consistent penetration into dentinal tubules than other sealers both *in vitro* and *in vivo*.¹⁵ Further microleakage has been found to be least in the case of resin based sealers.¹⁶ Cho *et al.* have also mentioned in their study that the bond strength of final restoration was the least affected in case of resin based sealers than the zinc oxide eugenol root canal sealer.¹⁷ Kim *et al.* have stated that resin based sealers (AH 26, EZ fill and AD Seal) are more biocompatible and have advantage in terms of radiopacity.¹⁸

In paint industries, solvents are often used to soften resin coating materials on paints to allow their easy removal.¹⁹ Thus solvents used in removal of paints can be considered in root canal retreatment for removal of strongly adhering resin based sealers from root canal walls.¹⁹ Chloroform and Xylene as a solvent for root canal sealer have been studied, but U.S. Food and Drug Administration has banned chloroform due to its potential for carcinogenicity and cytotoxicity.^{5,19-21} Use of D-Limonene (Refined Orange Oil) in endodontics is becoming popular due to its confirmed

biocompatibility, safety, and noncarcinogenic property, but Martos *et al.* and Mushtaq *et al.* have mentioned that the performance of orange oil as a solvent was inferior to xylene and chloroform.²²⁻²⁴ Because of concerns about Chloroform, clinicians and researchers have developed a renewed interest in finding an alternative solvent. Xylene is chlorinated hydrocarbon commonly considered as gutta percha solvent.²⁵ It may also soften or dissolve the sealers and could potentially facilitate their mechanical removal.¹⁹ Use of Endosolv-R for removal of resin based sealer has suggested by Cohen, Duncan and Chong.^{5,11} It contains 66.5 grams of formamide and 33.5 grams of phenyl ethylic alcohol.²⁶

In this study, only one surface of sealer was exposed to solvent to simulate the root canal conditions. Softening was defined as reduction in hardness that resulted from exposure to solvent.¹⁹ After 1 minute, Xylene was most effective against AH Plus (86.1%) followed by Adseal (65.1%) sealer and least effective against AH 26 (8.5%). After 1 minute, Endosolv-R was most effective against Adseal (69.2%) followed by AH 26 (62.5%) and AH plus (61.4%) ($p < 0.001$). After 2 minutes, Xylene was most effective against AH Plus (89.1%) followed by Adseal (75.1%) sealer but least effective against AH 26 (10.6%). These results are in partial agreement with the study of Kfir *et al.*¹⁹ After 2 minutes, Endosolv-R was most effective against AH plus (94.4%) followed by Adseal (91.9%) and AH 26 (87.3%) ($p < 0.001$). In other words, after 2 minutes of exposure Endosolv-R has been found to be a more effective short term softener for all the three sealers and its effect was more pronounced in case of AH plus.

Setting of epoxy resin sealers involves polymerization and cross linking of their monomers, resulting in 3D lattice.¹⁹ This set polymer is unaffected by saline or water. Hydrophobic organic solvents such as Xylene and Endosolv-R may have the ability to penetrate this 3D lattice resulting in swelling of the lattice and reduction in strength and hardness.¹⁹ Thus softening occurs that facilitates their removal by scrubbing effect provided by files.¹⁹ Ramzi *et al.* have stated that Endosolv-R combined with rotary files has most effectively removed filling materials from the root canals, especially in the apical third.⁶ Vranas *et al.* have reported that Endosolv-R has significant softening effect on resorcinol-formalin pastes after 2 minutes.^{4,25} Gambrel *et al.* concluded in their probe penetration study that softening effect of Endosolv-R after 20 minutes was superior to other tested solvents.^{4,26} Shokubinejad *et al.* mentioned that Endosolv-R does not affect the bond strength of newer obturation materials with root canal dentin whereas Laxmi Narayan *et al.* showed that Xylene causes significant reduction in enamel and dentin microhardness and thus may reduce the bond strength of newer endodontic sealers.^{27,28}

Also, by Occupational Safety and Health Administration

(OSHA) guidelines, Xylene causes irritation of eyes and mucous membranes, gastrointestinal distress and toxic hepatitis when ingested, chemical pneumonitis, hemorrhages in air spaces when inhaled, cytotoxic reaction when extruded periapically.^{5,20,29} However Chutich *et al.* have suggested that the amount of Xylene periapically extruded was too small to cause toxicity.³⁰ Even less information regarding biocompatibility of Endosolv-R is available and it has been suggested to have fetotoxic properties.³¹ However, results mentioned in this study may vary in *in vivo* conditions based on setting characteristics of sealer in root canal system and availability of solvents to the sealer in curved & ramified canals.

Conclusions

Within the limitations of this study, it can be concluded that after 2 minutes, Endosolv-R was a significantly more effective short term softener than Xylene for all the tested sealers, and thus Endosolv-R can be viewed as a better substitute of Chloroform for softening and removing of epoxy resin based sealers.

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