New Insight into the Dissolution of Epoxy Resin–based Sealers

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Abstract

Introduction: The purpose of this study was to evaluate the use of solvents that are not traditionally used in dentistry for the dissolution of an epoxy resin–based sealer and the effect of ultrasonic agitation (UA). **Methods:** The dissolution of the AH Plus sealer (Dentsply DeTrey, Konstanz, Germany) and the effect of UA in various solvents (eucalyptol, xylene, chloroform, EndoSolv R [Septodont, Cedex, France], EndoSolv E [Septodont], methyl ethyl ketone [MEK], and ethyl acetate) were quantified. The dissolving capacity was assessed by weight loss, Vicker microhardness, scanning electron microscopy (SEM), and X-ray diffraction (XRD). The results were compared with factorial analysis of variance using IBM SPSS Statistics 23.0 software (IBM, Armonk, NY), considering a 0.05 significance level. A preliminary *ex vivo* study was performed in extracted teeth, with MEK activated by UA as the final irrigation after mechanical removal of the filling material. SEM was used for assessing the cleanliness of the root canal walls. **Results:** The new solvent proposals, ethyl acetate and MEK, exhibited high dissolution ability, almost reaching chloroform. UA increased dissolution. Vicker values corroborated the dissolution assays. SEM and XRD revealed that solvents affected mainly the organic component of the sealer. *Ex vivo* results confirmed the immersion model findings. **Conclusions:** MEK and ethyl acetate proved to be excellent alternatives to chloroform or xylene solvents. MEK presented a high sealer dissolving ability in a short period, especially with UA, without the potential hazards of chloroform, suggesting it is a good approach to AH Plus sealer's dissolution empowered by UA. These results should encourage further studies in order to confirm their clinical relevance. *(J Endod 2017;■:1–6)*

Key Words

Epoxy resin sealer, microhardness, organic solvents, scanning electron microscopy, solubility

Significance

The complete removal of filling materials has been identified as crucial for periapical healing. The new proposals for solvent sealers could help reach this goal and improve the outcome of retreatments.

Nonsurgical endodontic retreatment requires the removal of endodontic filling material to ensure maximum root canal cleaning and bacterial load reduction, focusing on the recovery or maintenance of periapical health (1). However, the complete removal of filling materials, which are often attached to the canal walls and dentinal tubules, is almost impossible (2, 3). Although the efficacy of endodontic solvents on gutta-percha is well described, there is some controversy over their action on sealers.

Chloroform, eucalyptol, xylene, EndoSolv R (Septodont, Cedex, France), and EndoSolv E (Septodont) have been used to assist the removal of filling material (4–6). Although chloroform is recognized as one of the most effective solvents (6–8), it is classified as a group 2B carcinogen by the International Agency for Research on Cancer (9).

AH Plus (Dentsply DeTrey, Konstanz, Germany) is a 2-component epoxy resin–based sealer with low solubility (10) and high radiopacity (10) that provides an effective apical seal (11) based on a polymerization reaction of epoxy resin amines. It has been continuously used in comparative studies on the physicochemical, biological, and antimicrobial properties of root canal sealers (12, 13). Epoxy resins, the major constituents of AH Plus, are soluble in oxygenated compounds such as ethyl acetate (CH_3-COO-CH_2CH_3) and methyl ethyl ketone (MEK) (CH_3-CO-CH_2-CH_3) (14); they are easily obtainable and have a similar cost to those available commercially. MEK and ethyl acetate are colorless volatile liquids soluble in water that are commonly used as industrial solvents (15). The United States Environmental Protection Agency has categorized MEK in group D (not carcinogenic to humans) (16). Ethyl acetate is considered non-mutagenic with low toxicity and the United States Food and Drug Administration has classified it as GRAS (generally regarded as safe) (17).

Passive ultrasonic irrigation has been reported to improve the efficacy of irrigation solutions (18, 19), and its use in endodontic solvents has recently been suggested (4, 20). The aim of this investigation was to evaluate the dissolution of endodontic epoxy resin–based sealer AH Plus in solvents that are not traditionally used in dentistry, such as ethyl acetate and MEK, and the effect of ultrasonic agitation (UA).

Materials and Methods

The tested solvents were chloroform (Fisher Scientific UK Ltd, Leicestershire, UK), eucalyptol (Dentaflux, Madrid, Spain), xylene (Sigma-Aldrich, St Louis, MO), EndoSolv

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R, EndoSolv E, MEK (VWR International SAS, Fontenay-sous-Bois, France), and ethyl acetate (Fisher Scientific UK Ltd). Distilled water was used as a negative control.

Standardized stainless steel molds with a 7-mm diameter and a 3-mm height were cleaned with acetone and fixed on stainless steel blades. AH Plus sealer was prepared following the manufacturers' instructions and placed into the molds. Ten minutes later, the specimens were transferred into a chamber (IKA KS 4000 ic Control; IKA-Werke GmbH, Staufen, Germany) at a constant temperature of 37°C for 48 hours. The samples were removed from the molds and weighted on a digital analytical scale before being immersed in the solvents (initial weight, W_0). At room temperature, the specimens were submerged into each solvent (for 2 and 5 minutes). Afterward, the specimens were removed with tweezers, dipped in 10 mL distilled water for 10 minutes to neutralize the action of the solvent, blotted dry, and placed again in the chamber at a constant temperature of 37°C for 48 hours. The specimens were weighted again (postimmersion weight, W_f). The sealer dissolution was quantified as a percentage, considering the difference between the initial and the final weight, according to the following equation:

$$Dissolution\ \% = \frac{W_0 - W_f}{W_0} \times 100$$

The effect of UA was also studied using the protocol described previously; AH Plus specimens (n = 7) were immersed in 10 mL solvent at room temperature and then subjected to agitation in an ultrasonic bath (RETSCH Solutions in Milling & Sieving, Haan, Germany) to a frequency of 30 kHz for 2 and 5 minutes.

Vickers Microhardness

Microhardness was measured in fully set sealer samples after immersion for 2 minutes without UA in the most effective solvents (ie, chloroform, MEK, ethyl acetate, and EndoSolv E). EndoSolv R was also assessed because of its specificity for resin-based sealers. Vickers microhardness was calculated using the durometer Duramin (Struers A/S, Rodovre, Denmark) by applying a load of 10 gf for 15 seconds (n = 7). The mean hardness value was calculated after each solvent. The sealer samples not immersed in solvents were used as a control. Softness was considered as any reduction in hardness after exposure to the solvent.

Scanning Electron Microscopy and Energy-dispersive Spectroscopy

The surface characteristics of the sealer samples were studied using a Quanta 400FEG SEM (FEI, Hillsboro, OR). Previously, the specimens were coated with gold/palladium using an SPI Sputter Coater (SPI Supplies, West Chester, PA) to provide conduction. The examined specimens were sealer samples before (control) and after solvent immersion with and without UA. The solvents used were chloroform and MEK.

X-ray Diffraction Analysis

X-ray diffraction (XRD) analysis was performed using a Siemens D 5000 diffractometer (D8 Discover; Bruker AXS, Karlsruhe, Germany) with Cu-Kα radiation (λ = 1.5418 Å). XRD analysis was conducted with a scan range of 25°–40° (2θ) using a step size of 0.02° and a sept time of 2. Phases were identified using EVA software (Bruker, Coventry, UK). The examined specimens were sealer samples before (control) and after immersion in MEK with and without UA.

TABLE 1. Mean Weight Loss (± Standard Deviations) for AH Plus Sealer Samples Immersed in Various Solvents (2 and 5 Minutes) with and without Ultrasonic Agitation (UA)

	2 min			5 min			Total		
	Without UA	UA	Total	Without UA	UA	Total	Without UA	UA	Total
Control	0.00*	0.00*	0.00*	0.00*	0.00*	0.00*	0.00*	0.00*	0.00*
Chloroform	14.63 ± 3.78	29.32 ± 1.49	21.97 ± 8.11	15.83 ± 2.30	62.19 ± 4.81	39.01 ± 24.32	15.23 ± 3.07	45.75 ± 17.39	30.49 ± 19.79
EndoSolv E	2.10 ± 0.63	5.05 ± 1.83	3.58 ± 2.02	2.09 ± 0.44	8.68 ± 1.82	5.38 ± 3.65	2.10 ± 0.52	6.86 ± 2.57	4.48 ± 3.03
MEK	3.52 ± 1.82	24.84 ± 2.30	14.18 ± 11.24	10.67 ± 2.18	34.23 ± 5.39	22.45 ± 12.85	7.09 ± 4.18	29.53 ± 6.29	18.31 ± 12.57
Xylene	0.00*	2.66 ± 0.60	1.33 ± 1.44	1.87 ± 0.50	6.64 ± 1.48	4.25 ± 2.69	0.94 ± 1.03	4.65 ± 2.33	2.79 ± 2.59
Ethyl acetate	9.50 ± 1.50	10.24 ± 1.78	9.87 ± 1.63	10.54 ± 2.47	13.42 ± 0.62	11.98 ± 2.28	10.02 ± 2.04	11.83 ± 2.09	10.93 ± 2.22
EndoSolv R	0.00*	0.00*	0.00*	0.00*	0.00*	0.00*	0.00*	0.00*	0.00*
Eucalyptol	0.00*	0.00*	0.00*	0.00*	0.00*	0.00*	0.00*	0.00*	0.00*
Total	5.95 ± 5.76	14.42 ± 10.99	10.19 ± 9.70	8.20 ± 5.76	25.03 ± 21.55	16.62 ± 17.80	7.07 ± 5.83	19.73 ± 17.80	13.40 ± 14.64

MEK, methyl ethyl ketone.

*These results indicate the nonsolubility of the sealer independently of UA and immersion time.