

EVALUATION OF RESIN/SILICONE BASED ROOT CANAL SEALERS. PART I: PHYSICAL PROPERTIES

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The aim of this study was to evaluate the physical properties of EndoRez, Diaket, Epiphany, Roekoseal, Fibrefill, GuttaFlow, AHPlus, AH26 to that of a traditional zinc oxide based Kerr PCS sealers. Ten samples of each material were evaluated for flow, radiopacity, solubility and dimensional change tests according to ISO standards 2001-6876. The findings were statistically analyzed. Radiopacity and solubility of all sealers were in accordance with ISO standards 6876, except dimensional stability. However, all sealers had expansion values above the ISO requirements. Epiphany sealer showed the greatest expansion and the most flow rates under the given condition ($p < 0.05$). AH26 sealer was found to be the most soluble sealer ($p < 0.05$). Although AHPlus sealer was found to be the most radiopaque sealer, the difference was not significant between AHPlus and Roekoseal, Epiphany and Kerr PCS ($p > 0.05$). According to the result of this study, all root canal sealers showed acceptable properties due to ISO standards except dimensional stability.

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1. Introduction

One objective of root canal treatment is complete seal of the prepared canal system with root canal sealer in conjunction with a core material¹. Root canal sealers are intended to fill the irregularities between the dentinal walls and the core material, as well as the accessory and lateral canals^{2,3}. Root canal sealers should meet certain general requirements such as adequate flow and dimensionally stability, sufficient radiopacity, low solubility, qualified sealing ability and good biologic behaviour⁴. Physical characteristics of sealers could affect handling and clinical behaviour of the sealer, and this results, having an influence on the quality of the root canal filling⁵⁻⁷.

Solubility is an undesirable characteristic because it may cause microleakage and dissolution of biologically incompatible components in the root canal⁸.

Dimensional change affects the integrity of the bond between the sealer and the root dentin or core material⁷. The irregularities of the root canals and presence of accessory canals make the flow characteristic of special interest. Sealers should flow into irregularities and lateral canals of the root canals without causing periapical extrusion⁹.

An ideal sealer must have sufficient radiopacity in order to be able to distinguish it from surrounding structures, to identify if it is in the canal or extruded into periapical tissue and to assess the quality of the root canal filling¹⁰.

A number of tests have been described to assess the physical properties of sealers. These tests have been systemized by both International Standards Organization and ANSI/ADA standards^{11,12}.

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Zinc oxide-eugenol sealers are the most frequently used for many years. Despite a long history of successful usage, they have some disadvantages such as staining, slow setting time and solubility¹³.

Recently methacrylate resin sealers have been introduced, aiming to create “mono-block” by the adhesion of the sealer to the thermoplastic polymer cone and the dentin walls of the root canal. Although so many studies have been published about sealing and adhesion properties of resin based sealer¹⁴⁻¹⁶, there is a few study published about physical properties of these sealers.

Therefore, the aim of this study was to evaluate the flow, solubility, dimensional change and radiopacity of eight resin and silicone-based sealers in comparison with a conventional zinc oxide-eugenol based sealer by using the methods proposed in the ISO standards.

2. Materials and methods

Flow, radiopacity, solubility, and dimensional change of nine different root canal sealers were measured according to the standards of ISO recommendations for dental root canal sealing materials¹².

Nine root canal sealers were evaluated in this study. The detailed information about the source and composition of these root canal sealers are listed in Table 1. The sealers were mixed according to the manufacturer’s instructions. Ten repetitions were performed for each canal sealer and the result was obtained by calculating the arithmetic mean.

Table 1: List of the root canal sealers, their composition and manufacturers

Sealer	Composition	Manufacturer
RoekoSeal	Polydimethylsiloxane, silicone oil, parafin, hexachloride platinum acid, zirconium dioxide.	Roeko, Langenau, Germany
GuttaFlow	Polydimethylsiloxane, silicone oil, zirconium oxide, gutta-percha	Roeko, Langenau, Germany
Diaket	Zincoxide, bismuth phosphate, propionylacetophenone, copolymers of acetate, vinyl chloride, vinyl isobutyl ether, triethanolamine	ESPE, Seefeld, Germany
AH26	Bizmut oxide, methenamine, silver, titanyum oksit, bisphenol-A-diglycidylether	DeTrey/Dentsply, Konstanz, Germany
AH Plus	Epoxy resins, zirconium oxide, iron oxide, calcium tungstate, silicone oil	DeTrey/Dentsply, Konstanz, Germany
EndoRez	Urethane dimethacrylate resin, zinc oxide, barium sulphate, resin, pigment	Ultradent, South Jordan, UT, USA
Epiphany	Bisphenol-A-glycidyl dimethacrylate, polyethylene glycol dimethacrylate, ethoxylated bisphenol-A dimethacrylate, urethane dimethacrylate, barium sulphate, silica, calcium oxide, bismuth, pigment	Pentron, LLC, Wallingford, CT, USA
FibreFill	Benzoyl Peroxide, UDMA, HDDMA, BISGMA, PEGDMA, Proprietary Carboxylic Acid Functional Resins, Silane-Treated bariumborosilicate glasses, barium sulfate, calcium hydroxide, silica, pigment	Pentron, LLC, Wallingford, CT, USA
Kerr Pulp Canal Sealer	Zinc oxide, silver, resin thymol iodide, eugenol, canada balsam	Kerr Sybron, Romulus, MI, USA

Flow: The center points of glass plates are marked with an acetate pen. 0.05 ml of the mixed sealer was placed on that center point of a glass plate with a graduated disposable 1 ml syringe (Ayset Plastik Tek. Elek. San. As., Adana, Turkey). At 3 mins after inception of mixing, a second glass plate and a load of 100 g was placed centrally on the top of the sealer. After seven minutes, the load was removed without moving the top glass plate from its place. The maximum and minimum diameters from the center point of the compressed disc of sealer were measured in mm using a digital caliper (Mitutoyo Corporation, Japan). If the difference between these measurements was within 1 mm, the average of the diameters were recorded as the flow rate.

Radiopacity: Ten acrylic plates containing nine wells with the diameter 5 mm and 1 mm thickness and a socket for aluminum step-wedge were prepared. The wells were filled with freshly mixed nine different root canal sealers. Another glass plate was placed on the sealers to ensure that the excess sealer was removed. Plates were kept at 37 °C and 95 % relative humidity until completely set. An aluminum step-wedge with thickness varying from 1 to 12 mm (increasing 1 mm per step) had been placed on the plates for reference. Then each sample was radiographed by a dental X-ray machine (Trophy, Vincennes, France) operating at 70 kVp, 10 mA and 0.17 seconds. The object-focus distance was 30 cm. Standardized images were obtained using phosphor storage plates (PSPs) from the Digora (Orion Soredex, Helsinki, Finland) digital system. Mean grey values (MGV) of three different areas in each sample and each step of aluminum step-wedge were determined by an image program (Image ProPlus 4.0 Media Cybernetics Inc, Silver Spring, MD, USA). The average values were calculated for each material and aluminum step-wedge. A calibration curve was generated from the radiographic densities of corresponding aluminum steps for each sample by using a software (Curve Expert 1.3) Mean gray values of the materials were converted into equivalent mm Aluminum thickness by the means of formula derived from that curve.

Solubility: Moulds (diameter: 20 mm, height: 1,5 mm) filled with freshly mixed root canal sealers were placed on a glass plate covered with cellophane sheet. Another glass plate was pressed on the top of the sealers. Samples of Epiphany and EndoRez sealers were kept in the anaerobic chamber, and the other samples were transferred to regular chamber at 37 °C for 24 h. The samples were removed from moulds, weighed using an analytical balance (AND HM-200, A&D Company Limited, MA, USA) with a degree of accuracy of 0,001 g and placed in petri dishes. Then, they were immersed in 50 ml distilled water and kept in 37 °C for 24 h. The weight change of specimens was recorded. The samples were removed and dried on absorbent paper and reweighed. The difference between two weights was calculated.

Dimensional Change: Cylindrical teflon moulds having a height of 12 mm and an internal diameter of 6 mm were placed on a glass plate covered by a cellophane sheet. The moulds were filled with the freshly mixed sealers until a slight excess of material was observed at the top. Another glass plate wrapped in cellophane sheet was placed on top of the sealer. The mould and the plates were held together firmly in a C-clamp. Epiphany and EndoRez containing samples were transferred to the anaerobic chamber to allow setting process properly (16). Other samples were kept in regular chamber. All samples were kept in chamber at 37 °C for three times more than the setting time as recommended by the manufacturer. Regular surfaces were obtained by grinding the flat ends of the moulds by 600 grit wet sandpaper and then cylindrical samples (~12 mm height x 6 mm diameter) were removed from the moulds. The distance between the flat ends of each sample was measured by using a digital caliper with an accuracy of 0.01 mm (Mitutoyo Corporation, Japan). The samples were stored in distilled water at 37 °C for 30 days. They were dried and their length were measured again. The difference in length was calculated as a percentage of the original length. Mean of the ten replicates was recorded as dimensional change of the sealer.

The data were evaluated statistically using one way ANOVA and Tukey post hoc tests (p=0.05)

3. Results

Flow:

The mean values of flow measurements for all root canal sealers were shown in Table 2. The flow values varied from 4.03 mm (Kerr PCS) to 11.37 mm (Epiphany sealer). But statistically, only Kerr sealer showed significantly lower flow rate than the other sealers ($p < 0.05$).

Table 2. Flow rates of root canal sealers

Sealer	Flow (mm) (mean±SD)
GuttaFlow	7,96 ± 0,29
Roekoseal	6,31 ± 0,27
AH26	8,55 ± 0,27
AHPlus	9,50 ± 0,20
Kerr PCS	4,03 ± 0,23
Epiphany	11,37 ± 0,28
EndoRez	7,00 ± 0,34
Diaket	9,91 ± 0,38
FibreFill	6,35 ± 0,25

Radiopacity:

Table 3 shows the mean gray value (MGV) and equivalent aluminum thickness (mm) of each root canal sealer. Radiopacities were expressed in millimeters of aluminum and higher values represented greater radiopacity, ranging between 3,22 mmAl and 8,92 mmAl. All sealers possessed radiopacity above 3 mm of aluminum, complying with the ISO requirements.

Table 3. Radiopacity mean values of all sealers.

	Radiopacity(MGV) (mean±SD)	Radiopacity value (mm Al) (mean±SD)
GuttaFlow	170,66±12,15	4,61±1,21
Roekoseal	200,42±9,01	7,06 ±1,37
AH26	165,84±31,41	4,68 ±2,56
AHPlus	214,42±12,15	8,92±1,34
Kerr PCS	209,87±14,88	8,02±2,08
Epiphany	214,78±17,21	8,59±2,48
EndoRez	192,93±13,03	6,45±1,27
Diaket	149,42±8,84	3,22±0,62
FibreFill	178,36±8,74	5,16±0,87

Although AHPlus sealer was found to be the most radiopaque sealer, but statistically only difference was found between GuttaFlow, AH 26, EndoRez, Fibrefill and Diaket sealer ($p < 0.05$).

On the other hand, Diaket was found to be statistically less radiopaque than AHPlus, Roekoseal, Epiphany, EndoRez and Kerr PCS ($p < 0.05$).

Solubility:

Average solubility (%) with standard deviation for the sealers is presented in Table 4 . All sealers showed values within the limits of ISO standards (weight loss ≤ 3 % of mass).

Table 4. Solubility rates (%) of root canal sealers.

Sealers	Solubility (%) (mean\pmSD)
GuttaFlow	0,249 \pm 0,115
AH26	-0,617 \pm 0,151
AHPlus	0,012 \pm 0,013
KerrPCS	2,426 \pm 0,733
Epiphany	1,319 \pm 0,232
EndoRez	1,283 \pm 0,191
Diaket	0,110 \pm 0,055
FibreFill	1,689 \pm 0,552

AH26 samples gained weight when immersed in water and was less soluble than the other sealers ($p < 0.05$). Kerr PCS found to be was the most soluble sealer ($p < 0.05$). EndoRez, Epiphany and Fibrefill sealers showed similar solubilities which were significantly greater than GuttaFlow, Roekoseal, AHPlus, and Diaket sealers ($p < 0.05$). Statistically no difference was found among GuttaFlow, Roekoseal, AHPlus, and Diaket sealer ($p > 0.05$).

Dimensional Change:

The dimensional change rates (%) for each root canal sealer were presented in Table 5. None of the sealers conformed to the standards of ISO (shrinkage ≤ 1.0 %, expansion ≤ 0.1 %). Although all sealers showed some expansion, Epiphany exhibited the most ($p < 0.05$). AHPlus sealer showed the least expansion, but the difference was not significant between Roekoseal and Kerr PCS ($p > 0.05$).

Table 5. The dimensional change rates (%) of root canal sealers.

Sealers	D. stability (%) (mean±SD)
GuttaFlow	1,185 ± 0,986
Roekoseal	0,356 ± 0,274
AH26	1,800 ± 0,978
AHPlus	0,314 ± 0,075
KerrPCS	0,419 ± 0,298
Epiphany	8,010 ± 1,151
EndoRez	2,132 ± 0,194
Diaket	0,571 ± 0,117
FibreFill	1,688 ± 0,303

4. Discussion

ISO and ANSI/ADA^{11,12} have standardized some technological tests to investigate the physical properties of sealers. Assessment of the flow, radiopacity, solubility and dimensional change properties were evaluated using methods recommended by ISO in the present study¹².

AH26 and AHPlus are both epoxy resin-based sealers. It's indicated that, AHPlus possesses advantageous properties (such as lower solubility, less shrinkage, higher radiopacity and better biocompatibility and no formaldehyde release) than AH26¹⁷. In the present study, AHPlus showed to be more radiopacity and flow, and less expansion, compared to AH26 ($p < 0.05$). However AH26 was less soluble than AHPlus in 24 hour test period ($p < 0.05$).

Flow characteristic is the consistency of the mixed sealer enabling it to reach narrow irregularities, lateral canals and the apical foramen⁴. Besides the difficulty to control the material during placement into the canal, high flow property may also result in extruded material over the apical foramen, compromising periapical healing^{9,18}. Particle size, film thickness, temperature, rate of insertion, internal diameter of the canal, powder/liquid or paste/paste ratio and shear rate are the factors influencing the flow rate of root canal sealers^{5,19,20}. The flow measurements of the root canal sealers vary widely because of the differences in the methods used in the studies. Vermilyea et al.²¹ used rotational viscometry, whereas Weisman²⁰ used simulated ultrafine canal. ISO standards stated that when a load of 120 g applied on a sealer disc, the disc should have a diameter not less than 20 mm¹². The same method (with 100 g load) had been used in the present study and the sealer disc diameters range was between 4.03 mm-11.37 mm.

The variability of the results of flow studies also depends on powder/liquid or paste/paste ratio of the mixed material. For some materials very small changes in this ratio may cause a profound change in disc diameter¹⁹. Orstavik¹⁹ emphasized the need for manufacturers to provide measuring devices giving optimum powder/liquid or paste/paste ratio of root canal sealers. Most of the root canal sealers (Epiphany, Fiberfill, Roekoseal, GuttaFlow, EndoRez) were in two barrel automix syringes which were provided standard mixtures. The other sealers were mixed according to manufacturers directions.

New dual-cured resin-based sealers have been promoted with the desirable property of bonding strongly with both canal walls and the core filling material and creating monoblocks within the root canal²². From this point of view, flow is a desirable property for resin-based sealers. Epiphany sealer showed the highest flow rate when compared to the others. This would provide well-adapted fillings and facilitate the formation of monoblock. High flow rate of resin-based sealers might also prevent polymerization contraction stresses. Thus shrinkage stresses on the canal walls during polymerization would be minimized²³. The other sealers also showed acceptable flow rates in the present study.

The sealer must be radiopaque in order to detect the extension and the quality of the root canal filling. Beyer-Olsen&Orstavik¹⁰ established a standardized system to measure the quality of radiopacity. They used an aluminum step-wedge with 2 mm increments as a reference to determine the equivalent aluminum thickness of the studied materials. In literature usually, conventional radiographic films and optical densitometers were used to evaluate the radiopacity of filling materials¹⁰. However, in some studies, indirect method by converting the radiographs to digital images were also used instead of optical densitometer^{24,25}. Meantime, imaging directly with digital X-ray systems are time saving, consistent and the quality of the radiographic images are satisfactory²⁵. In the present study, digitally radiographed samples were evaluated with a image-analysis computer program. By digital radiography, standardization of the study was achieved easily. The images could be evaluated consistently by the software, without time consuming to stages of chemical processing of conventional films. Neither direct nor indirect digital systems are mentioned on the ISO standards, therefore a modified standard is needed for the radiopacity evaluation of root canal filling materials²⁶. Rasimick et al.²⁷ stated that the imaging technique could affect the measured radiopacity values of the materials. Barium and bismuth containing materials could have different radiopacities on film and phosphor store plates²⁷. Also differences could be found in the aluminum alloy of the step-wedge, exposure time, focal film distance, kVp, and mAs affects the radiopacity measurements of materials in situ.

The radiopacity of root canal sealers should be at least 3 mmAl, but excessive radiopacity of the material is not mentioned by ISO standardization¹². The radiopacity rates of all sealers used in the present study were consistent with ISO standards. Radiopacity of AHPlus was found to be 8.92 mmAl, which exhibited the highest radiopacity of all sealers in this study. This findings collaborate previous studies results^{28,29}. However no significant difference was found between AHPlus and Roekoseal, Epiphany and Kerr PCS ($p>0.05$). Radiopacity properties of AHPlus sealer was contributed to zirconium oxide and iron oxide content²⁹. Epiphany sealer contains silane treated barium borosilicate glass, barium sulfate and bismuth which provides radiopacity. The radiopacity of Epiphany in this study was found 8.59 mmAl similar to previous study result³⁰. Kerr PCS includes silver particles to improve the radiopacity. Beyer-Olsen &Orstavik¹⁰ found Kerr PCS more radiopaque than AH26 and Diaket sealer similar to our findings. Zirconium dioxide is the radiopaque ingredient for Roekoseal. Roekoseal has a radiographic density equivalent to 7.06 mmAl higher than the values reported in previous studies^{25,29}.

Solubility is an undesirable property for a root canal sealer because it can cause the sealer to release components that may be biologically incompatible and formation of gaps can affect the hermetic seal of the root canal filling negatively. Because the sealers are commonly used with a core material, solubility of sealers do not constitute a clinical problem. However the solubility of sealer can negatively influence long-term seal of the root canal filling¹³. Major differences in sealing ability of the root canal sealers over the time, could be related to the solubility of the materials. In a study, the apical sealing ability of Epiphany was reduced after 16 months of water storage and this finding was correlated with the high solubility of Epiphany.

According to ISO standards the solubility of root canal sealers shouldn't exceed 3% mass fraction¹². All the root canal sealers studied were within this limit of solubility. Epiphany and EndoRez showed higher values than standards in previous report²⁸. This might be because of hydrophilic nature of methacrylate resin-based sealers and the release of unreacted monomers²². These sealers require anaerobic conditions for polymerization and setting. An uncured layer has been observed in the aerobic environment¹⁶. In the present study, samples were placed in an anaerobic chamber to obtain polymerized sealer discs. The low solubility degree of methacrylate resin-based sealers might be a result of anaerobic conditions and optimal polymerization.

Both Epoxy resin-based sealers (AH26 & AHPlus) found to be the less soluble than other sealers in the present study. These finding is in agreement with the previous studies findings^{9,30}. However, different test mediums or periods might effect the solubility assessment results.

The solubility rates of sealers in this study are consistent with those of Orstavik³⁰ who found AH26 the least soluble with increased weight. The researcher found Kerr PCS more soluble than Diaket, N2, AH26, Procosol and Endomethasone. The stability of the epoxy resins and insolubility of silver powder and bismuth oxide provides insolubility of AH26. Instability of the zinc eugenolate chelate matrix on the other hand, is responsible for the increased solubility rate of Kerr PCS³⁰.

Most root canal sealers shrink or expand as a result of setting. Dimensional change studies are important to show the potential of sealers to provide desired hermetic seal and bonding core materials to the dentinal walls. ISO standards recommend a maximum shrinkage of 1% or expansion of 0.1 % of the measured sample length for root canal sealers¹². In the present study, expansion rates of all sealers were above those defined by the ISO. Water absorption of resin-based sealers during setting might explain the high expansion rates^{7,22}. However water sorption and expansion have belated beneficial effects, compensating for the stresses developed on the canal walls during polymerization shrinkage²². Hydrophilic methacrylate resin-based sealer Epiphany showed the greatest dimensional change with 8 % expansion in accordance with the results of Hammad et al.⁶ reported that polymerization shrinkage after setting is a serious problem for resin-based sealers. According to the present study results, all resin-based sealers showed extreme expansion rates. More extensive research should be conducted to investigate the long term dimensional characteristics of resin-based sealers.

Resin-based sealers are gaining popularity with enhanced adhesive properties. However there are limited studies on their physical properties. Further investigations are required to clarify the relevance of compositions and ingredients of sealers with the physical properties. Also investigations should be directed toward the clinical consequences of the physical properties for successful root canal treatment.

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