Assessment of microleakage in root canals sealed with an experimental endodontic composite

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This study compares the apical microleakage (ML) of an experimental amorphous calcium phosphate (ACP) endodontic sealer with the ML of a commercial material with similar resin composition (Resilon). The penetration of the methylene blue dye solution following 72 h immersion at 37 °C was measured in root canals of single-rooted human teeth treated with: 1) experimental primers or Resilon primer, 2) experimental ACP selaer or Resilon sealer, and 3) Gutta-perha or Resilon filler. The results contradict the reported ability of Resilon to provide an almost hermetic seal or perform better than gutta-percha. The ML values obtained with ACP sealant (equal to or up to 16 % lower than the ML values in Resilon groups) merit further evaluation of this experimental material.

Key Words: Amorphous Calcium Phosphate; Biomaterial; Microleakage; Polymeric Endodontic Sealant.

1. INTRODUCTION

Research in endodontics has for the most part been primarily focused on the development of the tools and materials most effective in facilitating root canal treatment. The majority of endodontic treatment principles originally evolved on the basis of trial and error; only recently have scientific methods been adopted that identify appropriate clinical strategies. The biological basis for the therapy has received comparatively little attention, and the advancement of biologically-based knowledge significant to clinical endodontics has been slow. Several controversial and as-yet unresolved biological issues relevant to endodontics were discussed in a recent review article (1) which specifically deals with disagreements regarding the management of pulpal exposures by caries in adult dentition, as well as the causes of and measures to control infections of the root canal system. The primary objective of root canal therapy is to introduce the appropriate placement of a seal between the root canal system and the periodontium. An ideal root-end filling material should not only seal the root-end cavity hermetically, but should also be biocompatible, non-toxic, insoluble in tissue fluids, non-resorbable, dimensionally stable, capable of inducing osteogenesis and cementogenesis, easy to prepare and use, sterilizable, radioopaque, inexpensive, and not susceptible to denaturing in the presence of moisture (2-4).

Root canal treatment traditionally involves mechanical preparation of the root canal system, debridement by antibacterial fluids and obturation with an inert material. The three main functions of obturation are to entomb any bacteria remaining within the root canal system, to stop the influx of periapical tissue-derived fluid from reentering the canal and to prevent the coronal leakage of bacteria (5). It is generally well-known that microleakage (ML) between the root canal filler and root canal walls may adversely affect the results of root canal restoration. The most widely taught and practiced gutta-percha techniques are far from optimal in creating a “hermetic seal” (6). It has been suggested that bioactive rather

TABLE 1.
The composition of dual cure UPHM resin used to fabricate ACP composite.
TABLICA 1.
Sastav dvojno inicirane UPHM smole korištene u pripravi ACP kompozita.

Monomers/components of the polymerization initiator systems Monomeri/komponente sustava za polimerizaciju	Acronym Skraćenica	Mass (%) Masa (%)
urethane dimethacrylate; uretan dimetakrilat	UDMA	47.2
poly(ethyleneglycol)-extended UDMA; uretan dimetakrilat s ugrađenim poli(etilenglikol)-om	PEG-U	29.1
2-hydroxyethyl methacrylate; 2-hidroksietil metakrilat	HEMA	16.8
methacryloyloxyethyl phthalate; metakriloiloksietil ftalat	MEP	2.9
bis(2,6-dimethoxynezoyl)-2,4,4-triethylpentyl phosphine oxide & 1-hydroxycyclohexyl phenyl ketone	1850 Irgacure	1.0
benzoyl peroxide; benzoil peroksid	BPO	2.0
2,2'-dihydroxyethyl-p-toluidine; 2,2'-dihidroksietil-p-toluidin	DHEPT	1.0

than biostable, inert materials may play a useful role in root canal sealing (7-13). These bioactive materials may also possess antimicrobial properties (8, 9) and facilitate stimulation of host cells (10, 11). Additionally, they can release ions(11) and promote precipitation(12) thus leading to an improved seal in aqueous milieu (13).

We have recently formulated dual cure (DC; light plus chemical) urethane dimethacrylate (UDMA)/ poly(ethyleneglycol)-extended UDMA (PEG-U)/2-hydroxyethyl methacrylate (HEMA)/methacryloyloxyethyl phthalate (MEP) resin (designated UPHM resin) based, bioactive ACP polymeric composites capable of attaining high degrees of double bond or vinyl conversion (DVC) accompanied with relatively high polymerization shrinkage (PS) and moderate levels of polymerization stress (PSS) (14). High DVCs attained in these materials suggest low leachability of the un-reacted monomeric species, and imply low probability of adverse cellular responses. Although the extensive shrinkage/stress seen in these composites might present a serious clinical risk and, ultimately lead to ML, the water uptake that softens and degrades the resin matrix (15) may also allow for the relaxation of residual stresses that develop within the matrix during the polymerization shrinkage (16), and reduce or close marginal leakage gaps (17). It has already been suggested that a significant hygroscopic expansion (HE) of DC UPHM/ACP composites may offset relatively high volumetric shrinkage reported for these composites (14). In this study, the objective was to assess the ML in these materials. Our working assumption was that the extent of ML in teeth sealed with the experimental ACP/UPHM composite will not exceed the apical leakage of teeth root canals sealed with the commercial sealant of similar composition. To test this hypothesis, series of dye penetration tests were performed in which the experimental ACP endodontic sealer was compared with the Resilon sealer for its ability to guard against apical leakage. When choosing the dye penetration method, we were aware of its generally high susceptibility to errors and the fact that both the endodontic research community and practicing clinicians typically do not relate the results of such studies to potential clinical outcomes. However, we also believe that findings of the preliminary dye penetration measurements, a method still praised by some researchers for easy visualization, clear reference points and contrast with the surrounding environment (18, 19), will be indicative enough to determine the direction of our future research and, ultimately, lead to recommending this new material for use in clinical environment.

2. MATERIALS AND METHODS

2.1. Fabrication of the experimental ACP sealant

The bioactive zirconia-ACP filler was synthesized as previously described in detail (20, 21). ACP was ground (14) (assigned g-ACP) and characterized (20-23) by X-ray diffraction (XRD; DMAX2000; Rigaku/USA Inc., MA, USA), Fourier-transform Infrared (FTIR; Nicolet Magna FTIR System 550; Nicolet Instrument Corp., WI, USA) spectroscopy, particle size distribution (PSD; CIS-100 Particle Size Analyzer; Ankersmid Ltd., Yokneam,

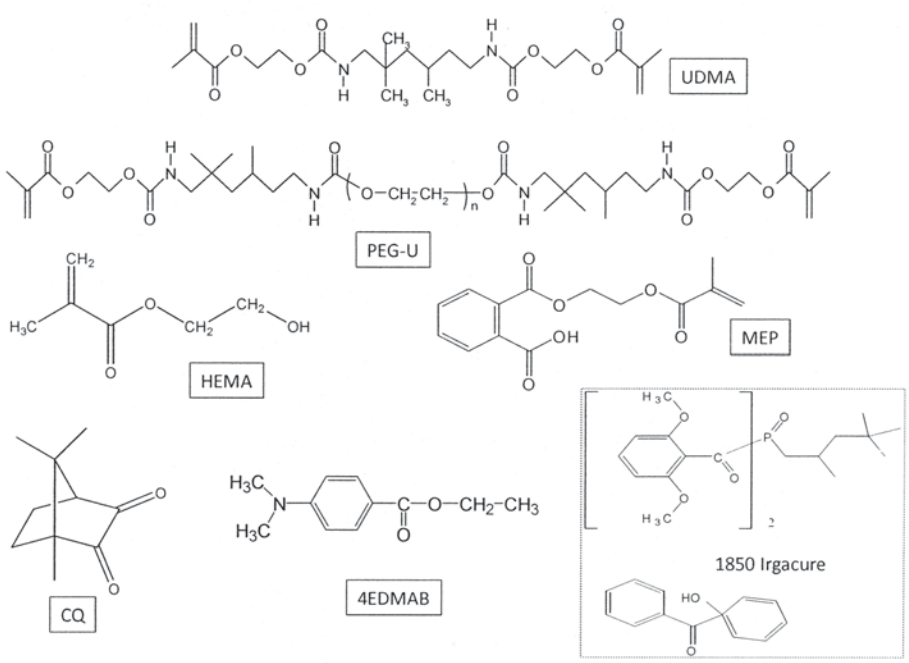

FIGURE 1.
Chemical structure of the monomers and components of the polymerization initiating systems employed to fabricate UPHM resin.
SLIKA 1.
Kemijska struktura monomer i komponenata sustava za iniciranje polimerizacije korištenih u pripravi UPHM smole.

Israel) analysis and scanning electron microscopy (SEM; JSM-5400; JEOL, Inc., MA, USA).

The resin was formulated from commercial urethane dimethacrylate (UDMA), poly(ethyleneglycol)-extended UDMA (PEG-U), 2-hydroxyethyl methacryalte (HEMA) and methacryloyloxyethyl phthalate (MEP) monomers to which photo- and chemical-initiators were added in proportions indicated in TABLE 1. Chemical structures of the monomers and components of the initiator systems are shown in FIGURE 1. After introducing the appropriate initiators to the monomer blends, the resin was stirred magnetically until it was fully homogenized. The dual-cure ACP/UPHM composite paste was prepared by mixing separately 1850 IRGACURE/BPO-containing UPHM resin and DHEPT-containing UPHM resin (mass fraction 60 %) with g-ACP (mass fraction 40 %), homogenizing the mixtures, and then combining the two pastes (1:1 mass ratio) to initiate chemical polymerization. The g-ACP easily blended at a 40 % mass fraction with UPHM resins and yielded highly flowable paste (FIGURE 2). The paste was applied to the prepared root canals and additionally light cured for 30 s at 450 mW/cm^2 (Dentsply curing unit, Milford, DE, USA). The physicochemical properties of the DC g-ACP/UPHM composites used in the study are compiled in TABLE 2.

2.2. The experimental protocol

The sequence of the preparative and measurement steps involved in the study is presented in **Figure 3.**

2.3. Preparation of teeth specimens

A total of forty-three single rooted human teeth were selected for testing (teeth with open apices, cracks and/or resorptive defects were excluded), cleaned with curettes to remove soft tissue remnants, and then stored in thymol-containing saline solution in refrigerator before instrumentation. The crowns of the teeth were sectioned at the cement-enamel junction using a water-cooled, low-speed diamond saw (Struers Minitom, Copenhagen, Denmark). The root canals were instrumented using a step-back technique. The instrumented root canals were irrigated with 10 mL 5.25 % solution of sodium hypochlorite and the smear layer was removed with 10 mL 17 % ethylenediaminetetraacetic acid (EDTA) solution following the protocol recommended in literature (24-26). Finally, the root canals were flushed with 3 mL 0.09 wt/wt % sodium chloride solution, dried with paper points and then randomly divided into eight experimental groups (5 or 6 teeth/group).

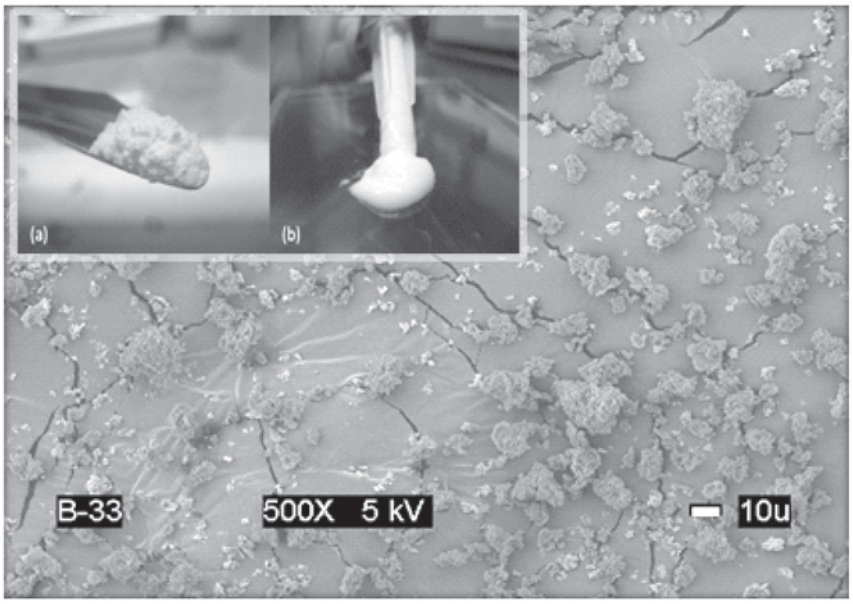

FIGURE 2.
Scanning electron microscopy image of the ACP filler used to prepare ACP/UPHM experimental sealer, and (inset) visual image of ACP filler (a), and the ACP/UPHM composite paste after mixing dual cure components (b).
SLIKA 2.
Pretražno elektronsko mikroskopska slika ACP punila korištenog za pripravu eksperimentalnog ACP/UPHM brtvila te (umetak) optičke slike ACP punila (a) i ACP/UPHM kompozitne paste nakon miješanja komponenti za dvojno iniciranje polimerizacije (b).

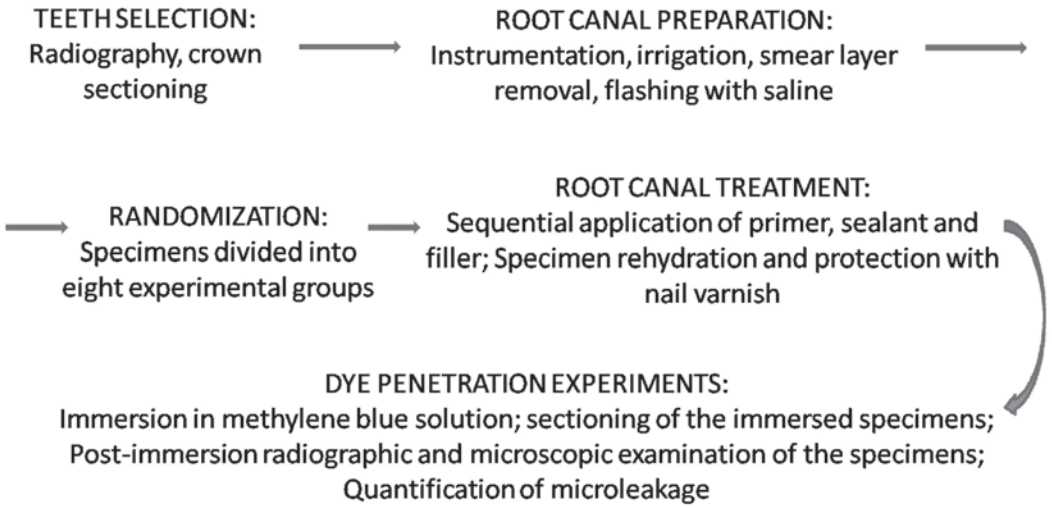

FIGURE 3.
Experimental steps involved in tooth specimen selection, preparation and dye penetration measurements.
SLIKA 3.
Eksperimentalni postupci korišteni prilikom odabira zubi, preparacije zubnog korijena i mjerenja prodiranja boje.

2.4. Root canal treatments (Figure 4)

The root canal walls of half of the specimens were primed sequentially with 1) 6.9 wt % solution of N-phenyliminodiacetic acid (PIDAA; Lancaster Synthesis Ltd., Windham, NH, USA; used as received, i.e., without purification) in acetone/water (50/50 wt % mixture), and 2) a multifunctional surface active monomer, the adduct of pyromellitic dianhydride and HEMA (PMDMA; 10 wt % solution in acetone containing also 0.5 wt % camphorquinone (CQ) to boost polymerization via photoredox reaction of CQ with PIDAA). The effectiveness of PIDAA and PMDMA as etchant/primer was documented earlier (27, 28). The other half of the speci-

TABLE 2.
Physicochemical characteristics of the experimental endodontic ACP sealer.[14, 21] Indicated are mean values ± standard deviation with the number of specimens in parenthesis.
TABLICA 2.
Fizičko-kemijska svojstva eksperimentalnog endodontskog ACP brtvila[14, 21] Navedene su srednje vrijednosti ± standardno odstupanje i broj uzoraka
(u zagradi).

Parameter	Value
Degree of vinyl conversion, DVC (%) Stupanj konverzije vinilnih funkcionalnih skupina	81.1 ± 2.4 (n = 3)
Polymerization shrinkage, PS (vol %) Skupljanje pri polimerizaciji	6.9 ± 0.1 (n ≥3)
Polymerization stress, PSS (MPa) Napetost uzrokovana polimerizacijom	3.6 ± 0.2 (n ≥ 3)
Water sorption, WS_{max}* (mass %) Adsorpcija vode	8.6 ± 0.5 (n ≥ 5)
Hygroscopic expansion, HE_{max}* (vol %) Higroskopna ekspanzija	12.7 ± 1.9 (n = 5)
Biaxial flexure strength, BFS[#] (MPa) Dvoosna savojna čvrstoća	41.8 ± 4.0 (n = 3)

*Maximum, plateau values obtained after 3 mo of aqueous immersion. [#]Wet specimens (immersed in buffered (pH=7.40) saline solution for 3 mo).
*Maksimalne (plato) vrijednosti u uzorcima uronjenim 3 mjeseca u vodu. [#]Mokri uzorci (uronjeni 3 mjeseca u puferiranu (pH=7.40) otopinu natrijevog klorida).

mens were primed with the commercial Resilon primer. The later comprised sulfonic acid terminated functional monomer, HEMA, water and the polymerization initiator. Resilon primer is a component of the new technology in endodontics, i.e., the Resilon-Epiphany system for root canal obturation (29, 30). The PIDAA/PMDMA primer system was applied in a following manner: first, two drops of PIDAA solution were placed in the canal to ensure the apex was reached, and then a micro-brush was used to coat the rest of the canal for 20 s. The canal was then dried for 2-3 s and any excess PIDAA solution was removed with a paper-point. The PMDMA solution was applied and the PMDMA coat was air-dried in the same manner. The Resilon primer was applied in one step, using a paper-point to completely coat the canal and subsequently remove any excess.

The root canal surfaces were sealed with the experimental dual-cure ACP/UPHM composite or Resilon sealer (comprised of dual-cure 2,2-bis(p-2'-hydroxy-3'-methachryloxypropoxy)phenyl propane (Bis-GMA), ethoxylated bisphenol A (EBPADMA), UDMA, hydrophilic difunctional methacrylate resin matrix and calcium hydroxide, barium sulfate, barium glass, bismuth oxychloride and silica as inorganic fillers). Resilon core material or gutta-percha were used as fillers for the following reasons: Resilon looks and handles like gutta-percha, it is available in standardized points that fit endodontic instruments, various tapers and accessory points and pellets for use with the Obtura II delivery system, and it can be placed in the canal by various techniques (single-cone, cold lateral condensation and thermoplastic method for example) using the same instruments that are utilized for gutta-percha application.[30] Both Resilon and gutta-percha fillers were applied by following the manufacturers' recommendations.

2.5. Dye penetration experiments

Once the filling process was completed, all but the apical 2 mm of each specimen were isolated with two layers of nail varnish and specimens were then rehydrated in saline solution at 37 °C for 48 h. Following the rehydration, specimens were immersed into 2 % methylene blue dye

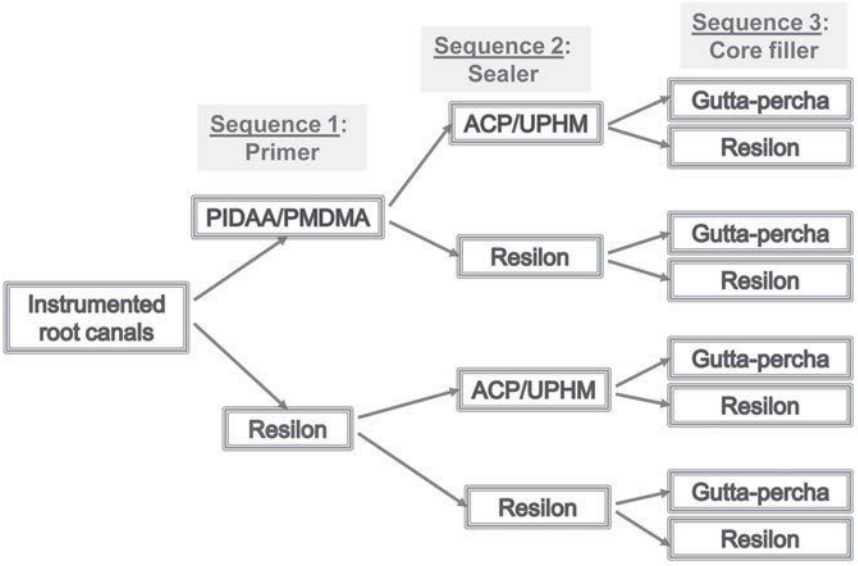

FIGURE 4.
Root canal treatment sequences. PIDAA/PMDMA solutions and ACP composite were prepared in our Laboratory. The Resilon-Epiphany kit was purchased from SybronEndo, Orange, CA, USA. Gutta-percha was procured from Obtura Spartan, Earth City, MO, USA.
SLIKA 4.
Slijed postupaka prilikom tretmana kanala korijena zuba. PIDAA/PMDMA otopina i ACP kompozit su pripravljeni u našem laboratoriju. Resilon-Epiphany je nabavljen od proizvođača SybronEndo, Orange, California, SAD, a gutaperka od kompanije Obtura Spartan, Earth City, Missouri, SAD.

solution for three days at 37 °C. Specimens were then removed from the dye solution, washed with distilled water and then dried. Finally, the teeth were sectioned longitudinally in a bucco-lingual direction through the center of the root (Struers Minitom, Copenhagen, Denmark).

2.6. Microleakage (ML) measurements

The linear apical leakage from the apex to the coronal extent of dye penetration was measured using the commercial digital-image-analysis system (Scion Image - release Alpha 4.0.3.2; National Institutes of Health, Bethesda, MD, USA) interfaced with an optical microscope (Olympus BX50F, Olympus Optical Co., Ltd., Japan) and digital camera (RGB/YC/NTCS; Microimage Video systems, Boyerstown, PA, USA). The microleakage value (ML) for each individual specimen was expressed as a distance (in mm) between the apical foramen and the 95 % unstained dentin boundary.

2.7. Statistical analysis

The experimental data were analyzed by using analysis of variance (ANOVA; $\alpha = 0.05$). The significant differences between the treatment groups were determined by all pair-wise multiple comparisons (Tukey test). The statistical calculations were performed by means of SigmaStat software (version 3.5; SPSS Inc., Chicago, IL, USA). For comparative purposes, one standard deviation (SD) is provided in this article as the estimated standard uncertainty of the measurements.

3. RESULTS

The g-ACP filler used to fabricate the experimental composite, showed two diffuse broad bands in $2\theta(^{o}) = (4 – 60)$ region (XRD spectrum), and a phosphate stretching absorption band at (1200 - 900) cm^{-1}, and a phosphate bending absorption band at (630 - 500) cm^{-1} (FTIR spectrum). Its particle size ranged from submicron up to 11.0 μm with the median diameter $d_m = (5.6 \pm 0.2)$ μm (PSD data). SEM images showing smaller agglomerates (≤ 20 μm) and prevalently fine particles confirmed the PSD analysis results. The results of the filler characterization are in accord with the previously reported structural and morphological characteristics of g-ACP (14). A diversity of dye penetration patterns is illustrated in FIGURE 5**.** The results of the ML measurements are summarized in TABLE 3. None of the eight treatment protocols provided a hermetic seal. Mean ML values showed no significant difference between treatments (one-way ANOVA). However, multiple pair-wise data analysis lead to the following conclusions: On average, root canals treated with the experimental ACP sealer leaked less than those sealed with the commercial products [(8.8 ± 3.4) mm

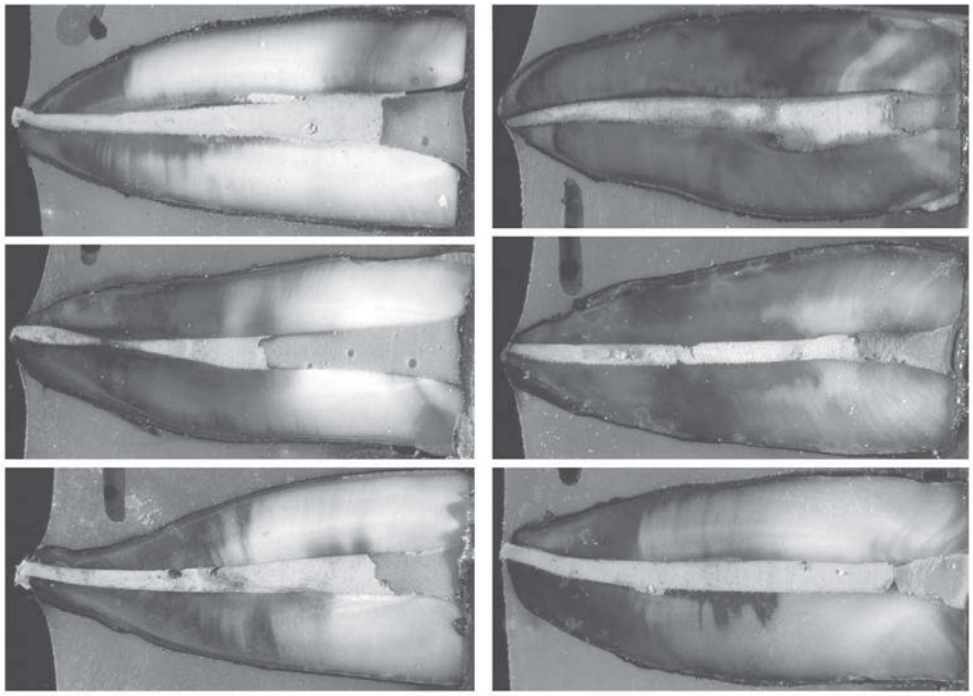

FIGURE 5.
Variability of dye penetration patterns observed in the study.
SLIKA 5.
Varijacije propuštanja boje zapažene tijekom experimenata.

TABLE 3.
Microleakge (ML; mean value ± standard deviation) for eight different treatment groups (as specified in FIG. 3). The number of specimens n ≥ 5/group.
TABLICA 3.
Mikro-propusnost (ML; srednja vrijednost ± standardno odstupanje) u osam različitih eksperimentalnih skupina kao sto je obrazloženo na SLICI 3. i broj uzoraka n ≥ 5 po skupini

Primer	Sealer	Filler	ML (mm)
PIDAA/PMDMA	ACP composite	Gutta-percha	5.8 ± 4.0
PIDAA/PMDMA	ACP composite	Resilon	9.0 ± 3.4
PIDAA/PMDMA	Resilon	Gutta-percha	9.4 ± 0.9
PIDAA/PMDMA	Resilon	Resilon	10.0 ± 3.8
Resilon	ACP composite	Gutta-percha	10.2 ± 2.1
Resilon	ACP composite	Resilon	9.8 ± 2.9
Resilon	Resilon	Gutta-percha	11.3 ± 5.6
Resilon	Resilon	Resilon	11.4 ± 4.9

vs. (10.5 ± 4.0) mm, respectively]. The treatments with experimental PIDAA/PMDMA primer and ACP sealer leaked the least distance from the apex. Treatments with at least two commercial components constituted the highest 12 % of leakages measured.

4. DISCUSSION

The curved, oftentimes complex shape of roots can make it difficult to achieve a desired curing efficacy with materials that utilize photo-initiation only. This problem can be counteracted by the secondary chemical curing that takes place in dual cure (DC) systems. For that rea-

son, g-ACP/UPHM composite intended for use as bioactive endodontic sealer was formulated as a DC system. The hydrophilicity of this experimental composite material stems from an affinity for water of both the resin matrix and the ACP filler (14, 20, 21). Physicochemical properties of this experimental material (TABLE 2) clearly indicate high polymerization efficacy of this dual-cure formulation (suggestive of very limited probability that the un-reacted monomeric species and degradation products would leak from the composite into the oral environment). Significantly, the relatively high PS in ACP/UPHM composites is likely to be compensated by a considerable level of HE when these composites are exposed to aqueous milieu (23). The beneficial effect of HE on PS has been documented for compomers and composites (16, 17) but could not be confirmed for contemporary posterior resin-based filling materials since their HE (on average 1.3 vol %) did not even equal the PS ((1.6 - 3.7) vol %) in these materials (31). In g-ACP/UPHM composites, the HE is almost twice as high as the measured PS (12.7 vol % vs. 6.9 vol %). Additionally, in UPHM composites fabricated with g-ACP sufficient paths for water diffusion existed thus ensuring a desired levels of bioactivity (mineral ions are released as a result of the intra-composite, water-catalyzed, transformation of ACP into thermodynamically stable apatite) and the potential for improved marginal and apical seal via precipitate formation at the sealer/tooth interface (13). Calcium and phosphate ion release from our experimental materials occurs at levels that significantly exceed the minimum necessary for the re-precipitation of tooth mineral (reported solution saturation ratio (SR) with respect to apatite SR >> 1.00) (23). Significantly, compared to the previously formulated ACP composites based on Bis-GMA or EBPADMA (19, 32), DC g-ACP/UPHM composites exhibited equal or higher remineralization potential, thus confirming their strong potential for preventing, or possibly even reversing root caries. Similarly to the experimental ACP sealer, the commercial control Resilon sealer is also hydrophilic in nature, and curing of the resin is achieved by both light and chemical initiation (29, 30). However, inorganic components of Resilon sealer are unlikely to create the supersaturation conditions conducive for the re-precipitation of tooth mineral at the sealer/etched tooth interface in a manner similar to the experimental ACP sealer. Only sodium and calcium ions are reportedly released from Resilon specimens and no distinct calcium phosphate precipitation could be detected on its surface (12). Therefore, if both materials were to exhibit comparable sealing efficacy, the experimental ACP sealant would have an advantage over Resilon sealer due to ability to act as an anti-caries agent.

The ML studies of various endodontic materials typically employ *in vitro* dye penetration, fluid filtration and/or bacterial infiltration methodologies (2, 26, 30, 33-40). Many of these studies produced quite contrasting results when comparing the same types of materials but using different screening techniques. In this study, we have used the dye penetration method as, in our judgment, a simplest way to compare the sealing potential of different materials. High ML values obtained irrespective of the type of the primer, sealant and/or filler used to complete the restoration of root canals, indicate that under given conditions (and within the limitations of the used methodology) a desired goal of attaining a hermetic seal could not be accomplished. Similar to our findings, a significant ML at the material/dentin interface and even penetration of the dye into the bulk of tooth specimens was reported for various restoratives tested via dye penetration method (26, 34-36), but also when less-error prone methods such as capillary flow porometry (37) or bacterial leakage (38, 39) were utilized. Particularly striking are the studies where the efficacy of gutta-percha and Resilon were found about the same (26, 37, 39), which is in sharp contrast to reports of superior Resilon performance (30, 40).

Exceptionally high ML values seen in all groups tested in this study are difficult to explain. It has been reported that in addition to being dependent on the location within the root canal (coronal, middle and/or apical) dye penetration systematically decreases with increasing tooth age (41). Also, the number of bacteria-invaded tubules and depth of bacterial penetration into dentin tubules of instrumented teeth is significantly higher in younger compared to older patients (42). High ML values obtained in our study could possibly be explained by assuming that the majority of teeth used in dye penetration experiments were collected from young patients having highly penetrable root dentine that hampered the effectiveness of the treatments. However, since the teeth obtained from the local dentists have no identifiers, our assumption cannot be confirmed. It can therefore be considered only as a speculative cause of the observed trend.

The possibility that inadequacy or inconsistency in the preparation of root canals was a major contributing factor to the extreme ML was circumvented by involving an experienced endodontist in specimen preparation. It is also unlikely that the inconsistencies in application of primers, sealers and/or fillers lead to the extensive leakage since the already established protocols were strictly followed in the study. One possible explanation for the teeth that were completely saturated with dye is that during specimen sectioning, the saw blade and cooling liquid coated the interior of the tooth with residual methylene blue.

Bacterial leakage tests performed with gutta-percha or a synthetic polyester-based endodontic obturation material that contains bioactive and radiopaque fillers, and calcium-hydroxide-based or epoxide/amine resin-based commercial sealers have shown that, while the rate of bacterial penetration along root canals may be affected by the presence (slower rate) or absence of smear layer (higher rate), the removal of smear layer did not impair bacterial penetration along root canal fillings regardless of the type of the sealer or filling material used (43). The irrigation and smear layer removal protocol (41) was identical to the one used in this study. It is therefore, unlikely that the "state" of the smear layer (possibly its incomplete removal) may have significantly contributed to high ML values measured in this study.

It has been documented (28) that PIDAA in acetone/water solvent performs two critical functions: (1) as dentin conditioner, it removes the smear layer and diffuses into conditioned dentin, while (2) as a primer/activator, it aids in the infiltration and initiation of polymerization of carboxylic monomers to form a polymer-reinforced dentin surface. PMDMA (27) promotes bonding to preconditioned dentin through easy diffusion into altered, porous dentin structure and the removal of some of the water from the micro-pores. There is no apparent reason as to why the priming procedure utilizing PIDAA/PMDMA or Resilon primer would fail in our experiments. The latter contains HEMA which is well-known for its hydrophilicity, excellent penetrability and the ability to form strong hydrogen bonds in the microporosities of pretreated dentin. Possible speculative explanation could be that with both treatments, the surface-active components of the primer (PIDAA and/or PMDMA in the experimental procedure vs. HEMA with Resilon primer) bonded weakly to the dentin which then resulted in equally weak (and porous?) primer/sealer and sealer/filler interfaces. To prove this hypothesis a comprehensive optical and/or scanning electron microscopy study should be performed. Alternatively, interactions of PIDAA/PMDMA and/or HEMA with dentin could potentially be examined using X-ray micro-computed tomography (μCT) (44). Both microscopic and μCT studies will be performed in the near future.

Another important property of root canal sealers, since these materials frequently come into contact with periapical tissues, is their biocompatibility. It has been reported that the biocompatibility of different material classes and products of root canal sealers vary considerably and the results are highly methodology-dependent (3, 45-52). The variability of the results obtained with practically all classes of materials accentuate the need for continuing research efforts in designing endodontic materials that combine sealing and bonding properties of resins with acceptable biological properties. Future evaluation of the experimental dual-cure ACP/UPHM composite will require additional ML assessment employing reportedly less error-susceptible methodology (fluid filtration or bacterial infiltration) to re-confirm or dispute the findings of this study. In addition, quantitative leachability measurements in conjuction with cytotoxicity tests appear necessary before this ACP-based experimental endodontic sealer could be recommend for testing in either animal model study and/or clinical trial.

5. CONCLUSIONS

Neither of the treatments involving the experimental primer/etchant system or Resilon primer, the experimental ACP sealer or Resilon sealer, and gutta-percha or Resilon as a core filler material provided a hermetic seal. Significantly, leakage in all treatments utilizing the experimental ACP sealer was equal to or lesser than leakage observed in commercial controls. Results of this study are in contrast with the reported ability of Resilon to provide seal to a greater degree than gutta-percha. Extended testing employing methodologies less prone to errors than the dye penetration method to re-confirm the findings of this study, combined with quantification and identification of leachables and cytotoxicity tests, need to be performed before this bioactive material could be recommended for use as endodontic sealer in clinical settings.

Disclaimer

Certain commercial materials and equipment are identified in this work for adequate definition of the experimental procedures. In no instance does such identification imply recommendation or endorsement by the American Dental Association Foundation or that the material and the equipment identified is necessarily the best available for the purpose.

Opovrgnuće

Komercijalni materijali i instrumenti identificirani u ovome članku navedeni su isključivo u svrhu pojašnjenja eksperimentalnih postupaka. Njihovo identificiranje ni u kom slučaju ne znači da su upravo ti materijali odnosno uređaji najbolji za opisana istraživanja niti ih Fundacija američkoga dentalnog udruženja i Nacionalni institut za standarde i tehnologiju u te svrhe izričito preporučuju.

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Appendix 1. List of acronyms; Dodatak 1. Popis kratica

ACP: Amorphous calcium phosphate; amorfni kalcijev fosfat
ANOVA: analysis of variance; analiza varijance
BFS: Biaxial flexure strength; dvoosna savojna čvrstoća
Bis-GMA: 2,2-Bis(p-2'-hydroxy-3'-methacryloxypropoxy)phenyl propane; 2,2-bis(p-2'-hidroksi-3'-metakriloksipropoksi)fenil propan
BPO: Benzoyl peroxide; benzoil peroksid
CQ: Camphorquinone; kamforkinon
DC: Dual cure; dvojno iniciranje polimerizacije smole
DHEPT: 2,2'-Dihydroxyethyl-p-toluidine; 2,2'-dihidroksietil-p-toluidin
DVC: Degree of vinyl conversion; stupanj konverzije vinilnih funkcionalnih skupina
EBPADMA: Ethoxylated bisphenol A dimethacrylate; etoksilirani bisfenol A dimetakrilat
EDTA: Ethylenediaminetetraacetic acid; etilendiamino tetraoctena kiselina
FTIR: Fourier-transform infra red spectroscopy; Fourier-transform infracrvena spektroskopija
HE: Hygroscopic expansion; higroskopna ekspanzija
HEMA: 2-Hydroxyethyl methacrylate; 2-hydroksietil metakrilat
HICR: Hybrid ionomer composite resin; hibridna ionomerna kompozitna smola
g-ACP: Ground ACP; usitnjeni ACP
1850 Irgacure: commercial photoinitiator system; komercijalni fotoinicijator

MEP: Methacryloyloxyethyl phthalate; metakriloiloksietil ftalat
ML: Microleakage; mikro-propusnost
MTA: Mineral trioxide aggregate; mineralni trioksid agregat
PEG-U: Poly(ethyleneglycol)-extended urethane dimethacrylate; uretan dimetakrilat s ugrađenim polietilenglikolom
PIDAA: N-phenyliminodiacetic acid; n-fenilimino dioctena kiselina
PMDMA: Adduct of pyromellitic dianhydride and HEMA; spoj piromelitičnog dianhidrida i HEMA-e
PS: Polymerization shrinkage; skupljanje pri polimerizaciji
PSD: Particle size distribution; raspodjela veličina čestica
PSS: Polymerization stress; napetost uzrokovana polimerizacijom
SD: Standard deviation; standardno odstupanje
SEM: Scanning electron microscopy; pretražna elektronska mikroskopija
SR: Saturation ratio; stupanj zasićenosti
UDMA: Urethane dimethacrylate; uretan dimetakrilat
UPHM: UDMA/PEG-U/HEMA/MEP resin; UDMA/PEG-U/HEMA/MEP smola
WS: Water sorption; sorpcija vode
XRD: X-ray diffraction; rentgenska difrakcija
ZOE: Zinc oxide eugenol; cinkov oksid euginol

PROCJENA MIKROPROPUSNOSTI ZUBNIH KORIJENA OBRAĐENIH S EKSPERIMENTALNIM ENDODONTSKIM KOMPOZITOM

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Izvorni znanstveni članak

SAŽETAK

U radu je uspoređena mikropropusnost (ML) experimentalnoga endodontskog kompozita temeljenoga na amorfnom kalcijevom fosfatu s mikropropusnosti komercijalnoga materijala sa sličnim sastavom smole (Resilon). Mjereno je prodiranje metilenskog plavila u zubima uronjenim u otopinu boje 72 sata pri temperaturi 37°C. Korijeni su zuba obrađeni: 1) eksperimentalnim primerima ili Resilon primerom, 2) s eksperimentalnim ACP ili Resilon brtvilom, te 3) gutaperkom ili Resilon punilom. Rezultati su u suprotnosti s tvrdnjama drugih autora da Resilon hermetički zabrtvljuje kanal korijena zuba i da bolje ispunjava kanal od gutaperke. Vrijednosti ML-a s ACP brtvilom (jednake ili do 16% niže od vrijednosti ML-a u skupinama s Resilonom) ukazuju da je vrijedno nastaviti testiranje ACP experimentalnoga kompozita za endodontsku uporabu.

Ključne riječi: Amorfni kalcijev fosfat; Biomaterijal; Mikropropusnost; Polimerno endodontsko brtvilo