REALIZAREA ŞI EVALUAREA PRIN TESTE *IN VITRO* A UNOR NOI SIGILANȚI ENDODONTICI EXPERIMENTALI
ACHIEVING AND EVALUATION BY IN VITRO TESTS OF NEW EXPERIMENTAL ENDODONTIC SEALERS

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The root canal sealers have the most important role, being responsable for the final sealing of the tooth root. The novelty elements of this study are represented by the endodontic sealing materials developed-the basic monomer, the coloured bioactive glass, hydroxyapatite in combination with the various metal oxide (all obtained in the laboratory), combining the organic monomers or the inorganic powders, as well as the selection of the appropriate ratio between the organic and inorganic phase and of the optimal polymerization system. All the physicochemical properties of the experimental sealers tested are in accordance with ISO 6876:2001 and the literature data.

Sigilanții canalului radicular au rolul foarte important, fiind responsabili pentru sigilarea finală a rădăcinii dintelui. Elementele de noutate ale acestui studiu sunt reprezentate de realizarea materialelor de sigilare endodontică: obținerea monomerului de bază, sticla bioactivă colorată, pulberile de hidroxiapatită în combinație cu diferiți oxizi metalici (toate obținute în laborator), combinarea monomerilor organici sau pulberilor anorganice, precum şi selectarea adecvată a raportului dintre faza organică şi cea anorganică şi a sistemului optim de polimerizare. Toate proprietățile fizico-mecanice ale sigilanților experimentali testați sunt în conformitate cu ISO 6876:2001 şi cu datele din literatură.

Keywords: *biomaterials, optical microscopy, endodontic sealers, composites, push out test*

1. Introduction

One of the factors that contributed to the development of the resin-based sealants was the recognition (observation) that gutta percha does not adhere to dentin or to any conventional sealant used, such as cement-based sealants like zinc oxide-eugenol (ZOE) and resins-based sealants (epoxy) such as AH-26 or AH Plus [1].

The use of a sealer during the filling of the root canal is essential for endodontic success. It not only increases the chance of achieving an hermetic sealing, but also serves as filling for the channel irregularities and minor discrepancies between the canal wall and core material. Almost 58% of the endodontic treatment failures were due to incomplete filling of the root space [2]. The sealers often migrate through lateral canals or dentinal tubules and may help the microbial control, blocking the access of the microorganisms left on the root canal wall or in dentinal tubules [3 - 6]. An ineffective obturation is often a prelude to an eventual failure.

Besides the materials and current filling techniques, it is necessary to review the cements that are essential for a good seal of the root canal. Firstly, it must be admitted that all endodontic sealants have some degree of solubility. Secondly it must be acknowledged that most endodontic sealants do not stick to the dentin walls.

The objective of this study is to produce two experimental resin-based root canal sealers and to show their properties using the tests: flow, solubility and push out.

The novelty elements in the formulation of the endodontic sealing materials used in this study are represented by the basic monomer, the coloured bioactive glass, hydroxyapatite powders in combination with various metal oxides (all obtained in the laboratory), combined with the organic monomers or the inorganic powders, the selection of the appropriate ratio between the organic and inorganic phase and of the optimal polymerization system.

2. Materials and methods

Formulation of the experimental root canal sealers. In Table 1 is shown the chemical composition of the two experimental sealers and of the commercial RealSeal SE sealer. Experimental materials are formulated in paste-paste system and dual polymerization system. The A- paste

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Table 1

The chemical composition of the experimental sealers and RealSeal SE (Sybron Endo / USA) sealer
Compoziţia chimică a sigilanţilor experimentali şi a sigilantului RealSeal SE (Sybron Endo / USA)

Samples code *Cod probe*	The organic matrix *Matricea organică*		The inorganic filler *Umplutura anorganică*	
E_T	33% Bis-GMA_{336} 5% PCL $_{diol}$ 34% TEGDMA 28% UDMA	Bis-GMA_{336} 2 ,2-bis-[p-(2'-hydroxy 3'metacriloxipropoxi) phenyl]-propane- synthesized in the "Polymer Composite" department ICCRR- UBB, Cluj-Napoca TEGDMA- triethylene glycol dimethacrylate (Aldrich) UDMA- urethane dimethacrylate (Aldrich); PCL diol- polycaprolactone diol (Aldrich) HEMA- Hydroxyethyl methacrylate (Aldrich)	10% Zn colored glass (iron oxide -600 to 1500 ppm) 30% HA-Zn 10% HA-Ti radiopaque agents: 40% ZrO_2, 10% YbF_3	HA-Zn- hydroxyapatite combined with 10% zinc oxide HA-Ti- hydroxyapatite doped with 5% titanium oxide Zirconium oxide (Merck) Ytterbium fluoride (Merck)
E_H	33% Bis-GMA_{336} 5% PCL $_{diol}$ 34% HEMA 28% UDMA			
RS	Real Seal SE (SybronEndo Orange, CA, USA)	Bis-GMA, ethoxylated Bis-GMA, UDMA, hydrophilic monomers, fillers: calcium hydroxide, barium sulphate, barium glass, silica		

contains: 0.7% N, N-dihydroxyethyl-p-toluidine (Aldrich), 0.5% camphorquinone (Aldrich), 1% dimethylaminoethyl methacrylate (Merck) and the B- paste: 1% benzoyl peroxide (Merck).

Bioglass with zinc was obtained by the conventional melting method.

Experimental glass oxide composition (Merck): SiO_2 -40%; 10% Al_2O_3; 10% B_2O_3; ZnO - 30%; -6% P_2O_5; Eutectic: E_1- 2% and E_2- 5%; chromophores- iron oxide (Merck) from -600 to 1500 ppm; ratio Al_2O_3 / SiO_2 = 0.25; melting point = 1380 ^{0}C; melting time = 480 minutes.

The chemical composition of the two eutectics is: E1- 25% NaF, 12% CaF_2, 63% AlF_3 (Merck) having a melting temperature of 700^{0}C, and for E2- 60% CaF_2, 40% AlF_3 (Merck) having a melting temperature of 840 °C. The time necessary for the formation of the eutectic melt is 30-40 minutes.

The vitreous glass mass in frit form was grounded in a ball mill with a capacity of 1 L in which we have introduced: dry frit, milling ball, absolute ethanol in a ratio of 2: 3: 4. The mill operated at 60 rotations / minute, and was maintained in this mode for 10 hours.The specific surface area (Brunauer–Emmett–Teller) of the zinc glass, colored with iron oxid, is 2.8 m^2 / g.

The synthesis of hydroxyapatite with ZnO was achieved by aqueous precipitation process using calcium oxide, orthophosphoric acid (Merck) and ZnO (Merck) as precursors [7].

The synthesis of hydroxyapatite with TiO_2 was achieved by aqueous precipitation process using calcium oxide, orthophosphoric acid (Merck) and TiO_2 (Merck) as precursors [8].

2.1. Flow test

The consistency of the material is an essential property of the endodontic sealers which should penetrate the dentinal tubules in order to achieve both a tight seal and a possible natural reconstruction around the periradicular tissue.

Method: (0.05 ± 0.005) ml of sealer paste are placed between two glass plates, 180± 5 seconds after beginning mixing, the seccond glass plate was placed on the top of the sealer folowed by the placement of a 100 g weight for obtaining a system with a total weight of (120±2) g. After 10 minutes from beginning mixing the weight and the glass plate were removed from the top of the sealer and the maximum and minimum diameter of the compressed disk was measured, in accordance with Section 7.2 of ISO 6876:2001.The allowable minimum diameter for the discs is 20 mm [9].

2.2. The solubility

The disk-shaped test specimens were obtained by mixing equal amounts of the two pastes of the root canal sealer. After mixing, a light flux was applied (10 seconds on five points on each of the two surfaces of the specimen) with a LED dental lamp. E (Guilin Woodpecker Medical Instrument CO., LTD.). The disk-shaped test specimens were obtained in the teflon mold having a diameter of 20.0 ± 0.1 mm and a height of 1.5 ± 0.1 mm. The solubility was performed in accordance with Section 7.7 of ISO 6876:2001 [9], using three sets of parallel samples of each material, each set containing two disk-shaped specimens. According ISO 6876:2001 a weight loss of less than 3%.is the accepted value.

Method:Two specimens are placed in the shallow dish (Petri dish) so that they do not touch and they remain undisturbed in the dish. Water (50 ±1) ml is added and the dish is covered. The dish is placed in the cabinet for a period of time (24 h, 14 day) and then the specimens are removed. The specimens are washed with 2 ml to 3 ml of fresh water, recovering the washings in the shallow dish.

The specimens are removed, than the water is evaporated from the dish without boiling. The dish is dried to constant mass at (110 ±2) ° C, than the dish is cooled in the desiccator to room temperature before each weighing (accurate to the nearest 0.001g).

The solubility (% weight loss of specimens) value was calculated using the formula (1)

Solubility % =(Mf-Mi)/Me x 100 (1)

Mf - the final weight of the Petri dish after evaporation at 110° C (without boiling);

Mi - the initial weight of the Petri dish before addition of the water;

Me - the sum of the initial masses of the two disk-shaped test specimens.

2.3. Push-out test

For this test three sets were selected, using extracted human molars, obturated with the materials from Table 1. Teeth were embedded in PMMA resin, and were cut with the biomaterial's cut sample machine, Microton Buehler- IsoMet 1000 .

2.4. Teeth preparation

For the present study were included a total of 30 teeth that were extracted for orthodontic, periodontal and prosthetic reasons, 4 weeks before the study. For the batch uniformity, we performed digital x-rays in two different angles. The teeth that had internal root resorption, calcifications, previous endodontic treatments or teeth that have been identified with more than one canal per root, were removed from the study.

As root canal filling material was used gutta-percha in combination with an experimental root canal sealer formulated for this study. For comparison we used the Real Seal sealer. The root canal filling technique used was warm vertical condensation.

The teeth were stored in the sodium hypochlorite solution with a concentration of 2.5%, at 100% humidity and a 37°C temperature for 7 days.

The samples were then placed in distilled water for 10 minutes, rinsed with ethanol for 15 minutes and placed in an autoclave at a constant temperature of 37°C for 24 hours, in order to dry them.

The samples, properly dried, were placed in resin blocks and left for 24 hours to complete the setting reaction of the resin.

Push-out method: a constant force was applied, by means of a cylinder- piston of 0.70 mm thick attached to a universal mechanical testing machine (Lloyd Instruments- LR5k plus), to each slice of tooth On the surface of the filler material, a constant load of 0.5 mm / min was maintained until failure, causing so shear forces at the interface sealant / dentin and at the interface sealant / core material. Failure forces were recorded for each section of the tooth.

The retention strength S [MPa] of the "monoblock system" of the tested tooth slice was calculated by dividing the release force F (N) at the side surface A (mm^2) of the filling segment: S = F/A.

Each tooth slice was numbered and examined on both sides, before and after the push-out test under the stereomicroscope (STEMI 2000 C Zeiss, Jena, Germany).

3. Results and discussions

3.1. Flow

The consistency of the material depends on the ratio liquid/powder, the viscosity of the organic phase (the nature of the monomers in the mixture) as well as the particle size of the powder. Hui-Min Zhou and colleagues have investigated five consecrated endodontic sealants. The mean diameter values of the discs, obtained after completion of the Flow test (in mm) using the same standard, were: BC sealer-23.1; MTA Fillapex, 24.9; AH Plus-21.2; ThermaSeal-21.3; PCS-23.1 and GuttaFlow-20.5 [10]. Gabriela Alexandra Marín-Bauza et al used 3 ml syringes for this test, and investigated 0.5 ml of each sealer. The average value of diameter discs, obtained for AH Plus sealant was 36.76 ± 3.04 mm [11]. In his study, Norberto Batista de Faria-Júnior together with collaborators obtained by the method given in ISO 6876:2001 the following Flow test values: Acroseal-21.24 mm, AH Plus 22.72 mm, ActiVGP 24.90 mm, Endomethasone N 18.76 mm and 25.15 mm Sealapex [12].

In Table 2 are presented the diameter values of the discs at different ratios liquid/powder. It aimed to establish the optimal ratio between organic phase (liquid) and anorganic phase (powder mixture).

Table 2

Flow test results / *Rezultatele testului Flow*

Samples code *Cod probe*	Ratio: liquid / powder *Raport: lichid / pulbere*	The mean diameter values of the discs [mm] *Valorile medii ale diametrului discurilor [mm]*
E_T	1 :1	14.0
E_H	1 :1	15.0
E_T	1.2 :1	20.5
E_H	1.2 :1	20.0
E_T	1.5 :1	22.0
E_H	1.5 :1	22.0

The liquid / powder ratio chosen after the Flow test for the following tests was 1.2/1. We managed to obtain thyxotrophic effect.

3.2. Solubility

From literature, D. Helvacioglu Yigit [13] gives the following percentage values for solubility of endodontic sealers: GuttaFlow 0.249; AH26 - 0.617; AH Plus 0.012; Kerr PCS 2,426; Epiphany 1.319; Endo Rez 1.283; Diaket 0.110 and respectively FibreFill 1.689.

Claudio Poggio [14] obtained for AH Plus sealer an average solubility of 0.32%, different values for different consecrated endodontic sealants, some even more than 3%, occur in other literature studies [15]. AH Plus shows a low solubility in the composition, probably due to the presence of the hydroxyethyl methacrylate [16].

Djurica Grga et al led a study on the solubility of three known sealers in Hank's solution. This study was led over a period of 28 days. At 24 hours, the percentage of solubility was: Acroseal -0.004; Apexit: 0.46 and AH Plus 0.11. After 28 days, the percentage of the solubility was: Acroseal -0.9; Apexit 1.5 and AH Plus 0.4 [17]. Also, studies that have been carried out about the solubility of endodontic sealers in artificial saliva are numerous [18].

Depending on the proposed objectives: monitoring the solubility of the endodontic sealers in time, the influence of pH on the degree of solubility, dimensional change, changes or alteration of specimen's surfaces after storage in water or other liquids, etc. [19 - 22] have been the subject of numerous specialized studies .

Figure 1 shows the mean percentage of solubility of the two experimental endodontic sealers after 24 hours and after 14 days of storage in distilled water. It can be noticed that, after 24 hours, the average percentage values of solubility for both experimental root canal sealers are very close.

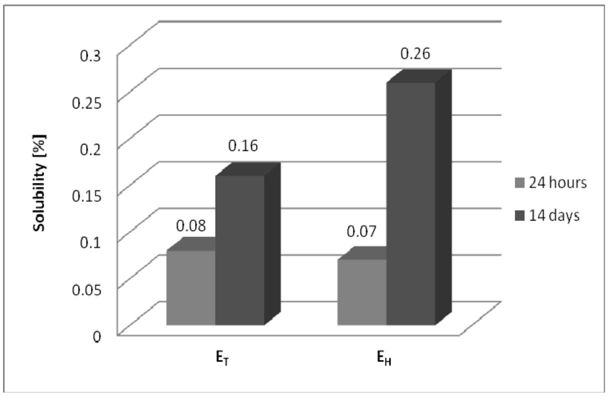


Fig. 1 - The mean percentage of solubility of the two experimental endodontic sealers after 24 hours and after 14 days of storage in distilled water / *Valorile medii procentuale ale solubilității celor doi sigilanți endodontici experimentali după 24 de ore şi după 14 zile de stocare în apă distilată.*

After 14 days, the value of the solubility E_T experimental sealer is significantly smaller than of E_H sealer. This finding can be explained due to the hydrophobic nature of the dilution monomer (TGDMA) from organic matrix of E_T sealer while the dilution monomer (HEMA) from organic matrix of E_H sealer has a hydrophilic nature, is able to absorb water, and therefore favors the solubility in time.

Finally, the percentage values of the two experimental root canal sealers are according to the requirements of 7.7 Section of ISO 6876:2001, lower than the maximum limits.

3.3. Push-out test

The push out test results show quite different values because it is very clear that the clinical conditions can not be reproduced. These differences may occur due to the fact that root dentine is not uniform and the root canal filling can be achieved by different techniques and different filling materials. Also, extracted teeth should be fresh and free from cracks. It is important to wash the canal with EDTA after using NaOCl, optionally followed by a final wash with sterile saline with 2% chlorhexidine, because oxygen left in traces of NaOCl inhibits the polymerization, forming a layer of residual monomer, which may lead to irritations [23 - 25]. Thorough cleaning of the root canal is indicated also for removing the smear layer.

It is also important to use for this test only teeth showing circular canals, if possible. The type of teeth used: molars, premolars, lead to significant differences in the results.

It would be good to fill a large number of teeth (10-20), which, as far as possible, should be first examined radiographically, in order to remove the cracked samples or the samples with too curved channels.

The teeth slices should have a thickness between 0.6 and 7 mm. Slices of 1 mm thickness are indicated in order to achieve a higher number of test samples [26].

In literature, differences are reported in the push-out resistance values between the specimen that used gutta-percha as core material or Resilon, although the used sealant was resin-based [27 - 31].

A. Jainaen and colleagues used for the push- out test teeth filled with gutta-percha / AHPlus, gutta-percha / EndoRez and Resilon / RealSeal. The push-out resistance values they reported were 2 ± 1.4 MPa (gutta-percha / AHPlus) and 0.4 ± 0.5 MPa for the other two systems [32].

Also, Emre Nagas et al carried out the push-out test for four systems of the endodontic sealing materials under different conditions of humidity of the root canal. He calculated the area of each section of tooth with formula $A = 2\pi rh$. The highest values for Push-out strength for all the blocking devices in the investigation were obtained when the root canal was dried under vacuum for 5 seconds followed by an application of the paper point for one more second. The highest value (over 3 MPa) was recorded for filling system: iRoot SP / gutta-percha [33].

Yasameen H. et al led a study to establish the adhesion by push-out test using different obturation techniques. For obturation system: gutaperca/AH26 they used lateral condensation, single cone technique and Thermafil technique and for the system: Resilon / Real Seal they used lateral condensation technique. Push-out test recordings were made at the apical, crown and mesial level. The highest values for all 3 levels were recorded for the gutta percha/AH26 combined with the technique of lateral condensation, 6.5; 6.8 and 7.2 MPa. For Resilon / Real Seal system combined with lateral condensation technique, the push-out resistance values reported are: 2.9 MPa for apical and mesial levels and 3.2 MPa for the crown level [34].

Tooth sections of 1 mm thickness were used for this test. The obturation technique used was the lateral condensation , respecting the clinical obturation protocol.

To avoid confusion between the slices of teeth, the slicing of all teeth was made from the apical to coronal direction. Each slice was numbered and examined on both sides of the tooth under the stereomicroscope (STEMI 2000 C Zeiss, Jena, Germany).

The teeth slices selected were the ones that were not damaged by cutting (detachment of one of the materials, cracks etc). As the root canal was considered to form a cone, and the tooth slices to have the shape of a truncated cone, we measured the diameter of each root canal section on each of the two sides of the slice, to be able to calculate the radius of base circle (R) and radius of the top circle (r). The lateral area of the canal section, in form of a truncated cone, having the formula $A_l = 2\pi\ G\ (R + r)$ [mm^2], where G is the slant height of the truncated cone in mm.

Each slice of the tested tooth was investigated on both sides with stereomicroscope at a 20x magnitude for noting the types of failure: failure at the sealer / dentin interface or at sealer / core material interface.

The average values of the retention force S, of the push- out test for systems with gutta-percha with two experimental sealants and gutta-percha with Real Seal sealer, are recorded in Table 3.

From the results of the push-out test (Table 3), the highest push-out strength was recorded in case of the system with gutta-percha / E_H experimental sealer and the lowest, for system with gutta-percha / Real Seal. E_H experimental sealer is a hydrophilic, dual-cured resin sealer containing urethane dimethacrylate and hydroxyethyl methacrylate. This might be attributable to the high affinity that sealer exhibits towards dentin. A good value of adhesion test was recorded for the gutta-percha / E_T system.

Literature data shows that the differences between the push-out resistance values might come from: the anatomy of the root canal in the region tested, the way the root canal was cleaned, the irrigating solution used, the system and technique used for filling the root canal, etc. [35 - 37]. *In vivo* the quality of adhesion on root canal dentine may be influenced by root canal irrigants and conditioners, root canal dentine hydratation as a result of pulp removal, and the polymerization degree of resin cement in root canals. In Figure 2. are presented some images obtained with the optical microscope at the medial level, before and after the push-out test for the tooth slices with the systems: gutta-percha / E_T, gutta-percha / E_H and gutta-percha / Real Seal.

From the images obtained with the optical microscope, after testing, we can observe a very good adhesion at sealer/dentin interface for all investigated sealants (about 70% of the investigated teeth). In addition, some 65% of the investigated teeth and sealed with E_H experimental sealers have a good adhesion at the sealant / core material interface. In case of the foreign sealer system only 40% of the investigated teeth had good adhesion at the sealer/ core material interface.

In at least 40% of the microscopically tested and investigated teeth, the failure was in the structure of the core material (gutta percha), or combined failures.

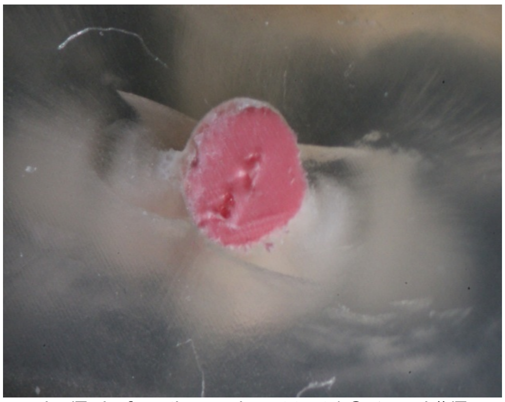
4. Conclusions

Two endodontic sealing materials were formulated and investigated by the tests: Flow,

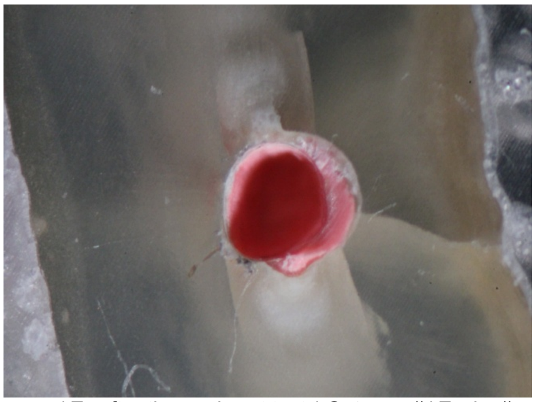
Table 3

Push-out test results / *Rezultatele testului push-out*

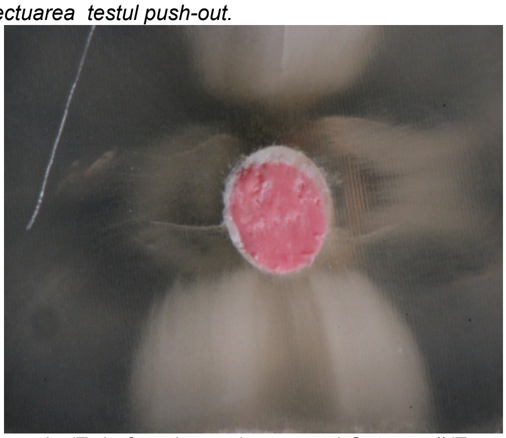
Series / *Seria*	I	II	III
Sealers / *Sigilanți*	E_T	E_H	Real Seal (dual)
Core material / *Material de umplutură*	Gutta-percha	Gutta-percha	Gutta-percha
Average push-out strength [MPa] at the apical level / *Rezistența medie push-out [MPa] la nivel apical*	1.9	2.7	0.9
Average push-out strength [MPa] at the medial level / *Rezistența medie push-out [MPa] la nivel mezial*	1.98	2.82	0.97
Average push-out strength [MPa] at the coronary level / *Rezistența medie push-out [MPa] la nivel coronar*	2.72	3.47	1.11



Gutta-percha/E_T before the push-out test / *Gutperchă/E_T înainte de efectuarea testul push-out.*

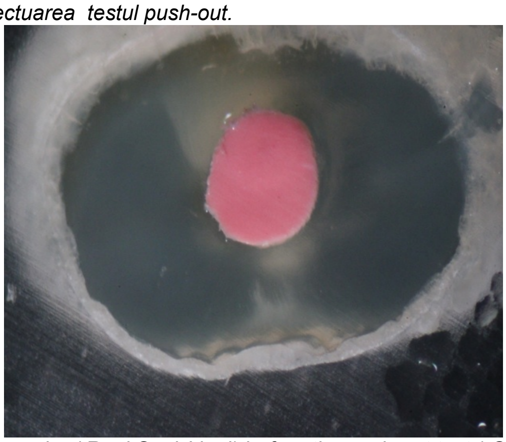


Gutapercă/ E_T after the push-out test / *Gutapercă/ E_T după efectuarea testului push-out.*

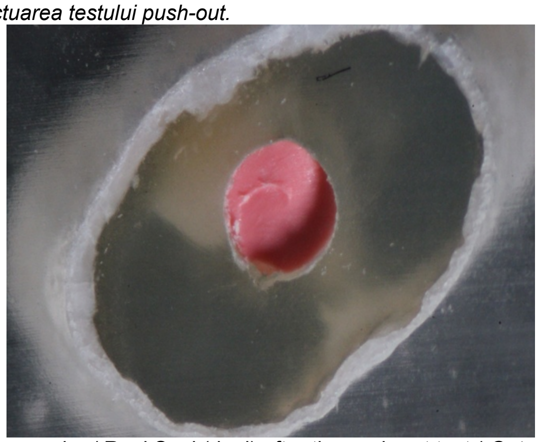


Gutta-percha/E_H before the push-out test / *Gutapercă/E_H înainte de efectuarea testul push-out.*

Gutta-percha / E_H after the push-out test / *Gutapercă/E_H după efectuarea testului push-out.*



Gutta-percha / Real Seal **(**dual) before the push-out test / *Gutapercă / Real Seal **(**dual) înainte de efectuarea testul push-out.*



Gutta-percha / Real Seal (dual) after the push-out test / *Gutapercă / Real Seal **(**dual) după efectuarea testului push-out.*

Fig . 2 - The images obtained with the optical microscope for the tooth slices with the systems: gutta-percha / E_T, gutta-percha / E_H and gutta-percha / Real Seal, before and after the push-out test / *Imaginile obținute cu ajutorul microscopului optic pentru feliile de dinți obturate cu sistemele: Gutapercă / E_T, gutapercă / E_H şi gutapercă / Real Seal, înainte şi după efectuarea testului push-out.*

solubility and push out. The results obtained from these tests lead to the following conclusions:

- Both experimental endodontic sealers have the desired consistency at the ratio liquid / filling of 1.2 / 1 by getting the thixotropic effect.
- After 14 days, the solubility percentage of the E_T sealer is lower than that of the E_H sealer. This finding may be explained by the hydrophobic nature of the dilution monomer (TGDMA) of the organic matrix from sealer composition of the E_T while the dilution monomer (HEMA) of the organic matrix from the E_H sealer composition has a hydrophilic nature and is able to absorb water over time. That led to a greater solubilisation of the components of the material.
- Highest push-out resistance in all three levels (apical, mesial, coronal) were reported for the system gutta-percha / E_H experimental sealer and lowest for gutta-percha system / Real Seal. A good adhesion value was recorded for the gutta-percha/ E_T system.
- All the physicochemical properties of the experimental sealers tested are in accordance with ISO 6876:2001 and the literature data for the other resin- based sealers.

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