Determination of *p*H and Evaluation of the Efficiency of Different Definitive Root Canal Filling Materials in Conservative Treatment of Chronic Apical Periodontitis

ANDREA CSINSZKA KOVACS IVACSON, FARAH CURT MOLA, CARMEN BIRIS, CRISTINA IOANA BICA*, MARIANA PACURAR, ADRIANA MONEA

University of Medicine and Pharmacy Tirgu-Mures, Faculty of Dentistry, 38 Gh. Marinescu str., 540139 Tirgu Mures, Romania

The phisico-chemical properties of the root canal filling materials play an important role in the outome of the endodontic treatment. Zinc oxide-eugenol, mineral-trioxid-aggregate (MTA), calcim-hydroxide or glass ionomer cement are the most frequently used chemical components of the materials, which assure them antimicrobial, radiopacity and adhesive properties. The high pH value ensures the sealer antibacterial effects which lead to the lowering of the periodontal inflammation and stimulate the healing process. The aim of this study was to determine in vitro the pH value of five of the most used root canal filling materials and to evaluate their efficiency in vivo in conservative treatment of chronic apical periodontitis. According to our results SealApex (Kerr) presents the highest average pH value and the shortest healing period of chronic apical periodontitis.

Keywords: root canal filling materials, pH, chronic apical periodontitis

Among a considerable percentage of the patients presenting chronic apical periodontitis conservative treatment-root canal treatment- can represent a therapeutic alterative. This therapy includes shaping, cleaning, and decontamination of the root canals and the obturation (filling) with an inert filling material [1]. In the healing process of the apical periodontitis among cleaning and shaping the root canal, the sealers (filling materials) also have an important role with their physical, chemical, antibacterial and biological properties. The producers of the endodontic filling materials are committed to respect the international regulations standards (ISO/DIS 6876 Dental root canal sealing materials; art. 57, American Dental Association) [2].

The researches are currently targeted in increasing the biocompatibility of dental materials that are in direct contact with the biological tissues [3]. Ideally, a dental material should contain no toxic, leachable, or diffusible substances that can be absorbed into the circulatory system, causing systemic responses [4]. According to Grossmann the ideal properties of the root canal filling material are the following: it should be easily introduced in the root canal system; seal the canal laterally and apically; not shrink after being inserted; impervious to moisture; bacteriostatic; radiopaque; not stain to tooth structure; not irritate periapical tissues; sterile and easily removable from the root canal if necessary [5].

The materials used for definitive obturation of root canal systems can be divided in groups based on the chemical elements found in their composition: zinc oxide-eugenol, mineral-trioxide-aggregate (MTA), paraformaldehyde, polyketone, epoxy, calcium-hydroxide, silicone, resins or glass ionomer [6]. The properties of each filling paste or sealer are defined by their chemical components. ZOE root canal filling pastes are the result of an acid based reaction between zinc oxide and eugenol with the formation of zinc eugenolate chelate [7] (scheme 1).

Zinc oxide is a well known semiconductor material that possesses several favorable properties, including wide

$ZnO+H_2O \rightarrow Zn(OH)_2$

 $Zn(OH)_2 + 2 HE \rightarrow ZnE_2 + 2 H_2O$

Scheme1. Setting reaction between zinc oxide and eugenol and the formation of zinc eugenolate chelate

band gap and large exciton binding energy (60 meV) at room temperature, good transparency or high electron mobility [8]. The zinc oxide pastes are cytotoxic, and may invoke an inflammatory response in the tissues. Though they are easily manipulative, radiopaque, germicidal and have good sealing properties [9].

Silicate binders also known as mineral trioxide aggregate (MTA), contain as main phases: calcium silicates (3CaO. SiO₂ and 2CaO. SiO₂) and Bi₂O₃ addition (necessary for increasing the radiopacity²); calcium aluminates (3CaO. Al₂O₃), calcium ferrite aluminates and calcium sulfate can be also found in small amounts in MTA composition [10]. MTA has excellent biological and physical qualities, evidenced by its antimicrobial effects, reduced cytotoxicity, stimulation of the cement depositing and periapical hard tissue formation. Recent studies have shown that MTA is similar to the Portland cement, but it contains 20% of bismuth oxide, with radiopacity effect. Through hydration, both of them produce hydrated calcium silicate gel and calcium hydroxide in a ratio of 4:1. This could explain the similar mechanism of action of MTA and calcium hydroxide [11,12].

Calcium hydroxides antimicrobial effects are due to the strongly alkaline properties given by the hydroxyl ions, which are released gradually. It can be used in combination with many substances, such as camphorated p-monochlorophenol, sterile saline, distilled water, anesthesic solutions, chlorhexidine, antibiotics, barium sulfate or iodoform .The major disadvantage of calcium hydroxide is its dissolving nature [12-15].

Glass ionomer cements result as a reaction between a silicate glass powder and a polyacrylic acid. The silicate cement assures the fluoride release and the translucency, while the adhesion to tooth structure is secured by the

^{*} email: cristina.ioana.bica@gmail.com; Phone: 0723180682

polycarboxylate cement compound [14,15] Setting reaction of glass ionomer cements is presented in figure 1.

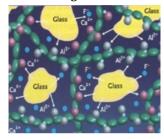


Fig. 1. Setting reaction of glass ionomer cements

A correctly chosen root canal filling material and a good technique, as well the experience of the doctor can increase the success rate after conservative therapy compared to surgical interventions. Although the surgical interventions present a higher success rate in the appropriate future, long term complications and even the loss of the teeth appear in 40-60 % of cases, in higher percentage than after endodontic treatment. This is the reason why more doctors choose the conservative treatment compared to surgical interventions [16,17].

The aim of this study is to determine in vitro the pH value of five of the most used root canal filling materials and to evaluate their efficiency in vivo in conservative treatment of chronic apical periodontitis.

Experimental part

Material and method

In this study, performed at the Faculty of Dentistry of Medicine and Pharmacy University of Tirgu-Mures, we evaluated, on the teeth of adolescent and adult patients, the *p*H value of 5 different sealers used on a wide range during endodontic therapy, the quality of the root canal filling and the evolution of the apical periodontitis, followed on periapical X-rays. The used sealers were Sealapex (Kerr), Endomethasone (Septodont), Endospad (Spad), MTA Fillapex (Angelus) and Endofill (PD). The chemical compositions of the used materials in research are presented in table 1.

The preparations followed the instructions of the producer. 0.5 g of material was added to 30 mL solution of KCl 0.1 N and was agitated for 5 min. For the registration of the pH values the Jenway 3320 pH-meter (fig. 2) was used at different time intervals, immediately after preparation, after 1 h, after 48 h, after 7 days and after 14 days.



Fig. 2. The Jenway 3320 pH meter

To demonstrate that the in vitro obtained *p*H value is similar to the results of the clinical cases, 40 adolescent and adult patients with radiological diagnosed periapical lesions were selected. Patients having systemic diseases or treated with antibiotics over the last 3 months were excluded from the study. Root canals were prepared with hand files (Flexofile, DentsplyMailleffer) using the stepback technique and enlarged in correlation with the diameter of the apical foramen. 2.5 % sodium hypochlorite was used as irrigant. After shaping and cleaning the canals, calcium hydroxide paste (Calxyd, Spofa Dental) was used as local intracanal medication for 10-14 days to decrease inflammation. Tooth crown was filled with glass-ionomer cement (Kavitan, Spofa Dental) to obtain a good isolation of the root canals.

NSAID were administrated orally for 3-4 days. Systemic antibiotics were not used since these medicines provide no benefit over localized infections.

On second appointment the cement was removed with diamond burs. The calcium hydroxide paste was cleaned with abundant irrigation and hand files.

The conditions to proceed the definitive canal obturation were the absence of pathologic symptoms and the possibility to dry the root canals. After cleaning and shaping, the root canals were filled with the 5 named sealers, 8 teeth with each material. Root canals were dried with paper points (Dentsply) and filled with the mentioned sealers and gutta-percha cones (Dentsply), using the lateral condensation technique.

Results and discussions

The found *p*H-values immediately after preparation after 1 hour, 48 h, 7 and 14 days for the five dental filling materials included in research, are shown in figure 3.

The found average of *p*H-values of the five sealers included in research, are presented in figure 4.

The correlation between the average pH-values of the materials and the healing of the chronic apical periodontitis



Fig. 3. The founded pH values of the five filling materials at the different time intervals

Table 1				
THE USED MATERIALS IN RESEARCH AND THEIR COMPONENTS				

Material	Composition		
SealApex	20% calcium oxide, 2.5% zinc oxide, 29% bismuth trioxide, 3% silicon particles, 20%		
(Kerr)	titanium dioxide, 1% zinc stearate, 3% tricalcium phosphate, isobutyl salicylate +		
	methyl salicylate + 39%, pigment		
Endomethasone	Hydrocortisone acetate 1.0 g Excipients: thymol iodide, barium sulfate, zinc oxide,		
(Septodont)	Magnesium stearate q.s.ad. 100.0 g		
Endospad	Diiodtimol, Enoxolan, Zinc-oxide, Ag. powder		
(Spad)			
MTA Fillapex	Salicylate resin, diluting resin, natural resin, bismuth trioxide, nanoparticles of silica,		
(Angelus)	MTA, pigments		
Endofill (PD)	0.01% dexamethasone acetate, 1% hydrocortisone acetate, 2.2.% polyoximethylene,		
	22.5% thymol iodide + eugenol liquid		



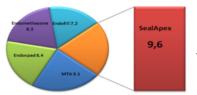


Fig. 4. The average *p*H values of the five sealers

lesions demonstrated on radiologic examination, are shown in table 2.

Table 2CORRELATION BETWEEN THE AVERAGE *p*H-VALUE AND THEHEALING PERIOD OF THE CHRONIC APICAL PERIODONTITIS

	Average pH- value	Healing period (month)
Sealapex (Kerr)	9.6	12-15
Endomethasone (Septodont)	8.3	14-18
Endospad (Spad)	8.4	12-18
MTA Fillapex (Angelus)	9.1	12-16
Endofill (PD)	7.2	16-20

We present below the radiological images (retroalveolar X-rays) of five patients included in the study, who received endodontic therapy with the previously mentioned dental materials.

Clinical case 1: Patient H. V., age 30, visits the clinic with pain at tooth 4.6. At clinical examination the tooth presents a massive coronal destruction, on radiograph it observes a periradicular lesion on the mesial root of the tooth. After cleaning and shaping the root canal is filled with Sealapex and Gutta Percha cones. After radiologic evaluation we concluded that Sealapex has the fastest action on the lesion, the approximate healing time is 12-15 months (fig. 5). After the radiologic evaluation, we concluded that this filling material present the lesions healing period between 14-18 months.



Fig. 5. A) OPG of the patient: a periradicular lesion can be observed at tooth 4.6; B) Rx. after 6 months; C) Rx. after 12 months

Clinical case 2: Patient C.P., age 30, comes to the clinic presenting pain at 4.7. Clinical examination shows a massive coronal destruction. On radiographs we observed chronic granulomatous apical periodontitis. After cleaning and shaping, root canals are filled with Endomethasone sealer and Gutta Percha cones. The radiological images (initial, after 6 months and after 12 months) are presented in figure 6. After radiologic evaluation we concluded that Sealapex has the fastest action on the lesion, the approximate healing time is 12-15 months.



Fig. 6. A) Initial radiograph of 4.7; B) Rx after 6 months; C) Rx after 12 months

Clinical case 3: Patient S.M., age 24 visits the clinic for oral rehabilitation. During radiographic examination we observed that the tooth 3.6 presented a chronic apical periodontitis. After cleaning and shaping, the root canals were filled with Endospad and GP cones. Endospad was

effective in healing the lesion, presented a healing period of 12-18 months (fig. 7).

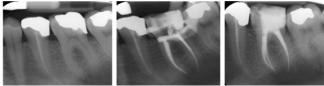


Fig. 7. A) Initial radiograph of 3.6; B) Rx after 6 months; C) Rx after 12 months

Clinical case 4: Patient S.D., age 26, visits the clinic because present a diffuse pain, which irradiate on the tract of the mandibular nerve. Based on the radiologic examination, the diagnosis was diffuse periradicular periodontitis which involved the roots of the tooth 4.6. After cleaning and shaping, root canals were filled with Fillapex-MTA and GP cones. Radiologic evaluation after 12 months showed a 90% healing of the lesion. Healing period was between 12-16 months (fig. 8).



Fig. 8. A) Initial Rx of 4.6; B) Rx after 6 months; C) Rx after 12 months

Clinical case 5: Patient B.P., age 20, visits the clinic presenting pain at tooth 1.5. Radiologic examination shows a periapical granuloma. After cleaning and shaping, root canal was filled with Endofill. This sealer has a slower action, and the complete healing period was 16-24 months (fig. 9).



Fig. 9. A) Initial radiograph of 1.5; B) Rx after 6 months; C) Rx after 12 months

The obtained results are similar to other data in the international literature. The high *p*H value ensures the sealer antibacterial effects which lead to the lowering of the periodontal inflammation and stimulate the healing process. One of the most important roles of the sealers with increased *p*H level is that they create optimal conditions for the alkaline phosphatase to act on periodontal tissues and stimulate the bone remineralization [18]. Alkaline phosphatase is a homodimeric protein enzyme, containing two zinc atoms crucial to its catalytic function per monomer, is optimally active at alkaline pH environments and has the physiological role of dephosphorylating compounds [7]. Contrary, the endodontic sealers with low *p*H-values increase the activity of the osteoclasts and induce bone resorption [19,20].

The antimicrobial activity of calcium hydroxide-based materials, such as SealApex may be related to ionization with subsequent release of hydroxide ions and an increase of *p*H levels, creating an unfavorable environment for microbial growth [21]. The release of hydroxyl- ions alters the integrity of the cytoplasmic membrane and causes biochemical injuries to the organic components. Since the action site of hydroxyl ions released from calcium hydroxide includes the enzymes in the cytoplasmic membrane, this

medication has a large scope of action depending on the amount of material, and therefore affects a diverse range of microorganisms, irrespective of their metabolic capacity. Therfore, we recommend the usage of sealers with higher *p*H-values especially in extended chronic periapical lesions. In case of moderate or small lesions sealers with lower *p*H values, as Endomethasone, Endofill or Endospad can be used with success.

Endomethasone is a frequently used sealer, indicated mainly in small periradicular lesions. After the researches of Suzuki [22], the limiting of the filling material to the root canal space apically is important to determine the best treatment outcome.

Many researches have also studied the properties, specially the *p*H-value of the root canal sealers [18]. Eldeniz et al [23] proved that Sealapex has a higher *p*H value compared with Apexit and Acroseal. Duarte et al [24] also found higher *p*H value for Sealapex comparing the mentioned sealer with Sealer 26 and Apexit. Vivan et al [25] demonstrated that sealers MTA Fillapex (Angelus) and MTA Bio present higher *p*H values compared with epoxidic resins based sealers or Portland cement. Massi and al [26] found that MTAS, an MTA-based sealer, presents high *p*H value, with highest *p*H recorded at an interval of 48 h. According to the international literature, data highest *p*H values are recorded for Sealapex, Sealer AH-26 or Apexit [27-29].

Without a rigorous cleaning and shaping of the root canal with carefully selected instruments and a good technique associated with irrigants, the root canal filling becomes indifferent and its' *p*H value has no significance [30-33].

Conclusions

The different *p*H-values of the sealers determined with the Jenway 3320 pH-meter demonstrate that each material has a different action period in time. The differences are due to the materials different physic and chemical compositions.

Even if the results were obtained in vitro, radiologic examination and follow-up of patients treated with the analyzed sealers, in case of extended periodontal lesions the use of root canal sealers with increased *p*H in strongly indicated.

The sealers with the highest *p*H values were Sealapex and Fillapex-MTA. These materials can be used in extended lesions, because the microorganisms are not capable of surviving in alkaline medium.

The other sealers- Endomethasone, Endofill, Endospadare recommended to be used in root canal treatment on teeth presenting moderate or small periapical lesions.

Periapical radiographs demonstrated that the time interval necessary for the resorption of the periodontal lesions is the shortest in case of sealers with increased pH values.

Among the used sealers, Sealapex (Kerr) had the highest *p*H value and the shortest healing period according to X-ray evaluation.

The root canal sealers which had calcium-hydroxide in their composition presented higher pH values and increased alkalization capacity of the endodontic and periapical space. Among these materials Sealapex showed the best results.

References

1.TORABINEJAD M, WALTON RE, Ed Saunders, 2008 2.COHEN S, HARGREAVES KM, Missouri, Mosby 2006. pp. 724–85 3.BECHIR, E.S., BECHIR, A., GIOGA, C., MANU, R., BURCEA, A., DASCALU, I.T., Mat. Plast., **53**, no.3, 2016, p. 394 4.HANCU, V., COMANEANU, R.M., COMAN, C., FILIPESCU, A.G., GHERGIC, D.L., COTRUT, M.C, Rev. Chim. (Bucharest), **65**, no. 6, 2014, p. 706

5.CASTELUCCI, Endodontics, vol. II, III, 1990, Ed. Il Tridente 6.BECHIR A, BECHIR E, Ed. Printech, 2012, pp. 34-40, 110-117

7.*** https://en.wikipedia.org/wiki/Endodontics

8.TABACARU, A., MUSAT, V., DINICA, R.M., GHEORGHIES, C., Rev. Chim. (Bucharest), 67, no. 8, 2016

9.MOTOC, O., POPOVICI, R., ONISEI, D., PODARIU, A.C., Rev. Chim. (Bucharest), **66**, no. 7, 2015

10.VOICU, G., BADANOIU, AI., ANDRONESCU, E., BLEOTU, C., Rev. Chim. (Bucharest), **63**, no. 10, 2012

11.MONEA., M., STOICA., A., BECHIR., E.S., BURCEA, A., PANGICA, A.M, Mat. Plast., 53, no.1, 2016

12.ESIAN, D., MAN, A., POP, S., EARAR, K., BUDACU, C.C., CERGHIZAN, D, BUD, A., BICA,, C., Rev. Chim. (Bucharest), **67**, no. 6, 2016

13.MARÍN BAUZA GA, SILVA SOUSA YTC , CUNHA SA, RACHED JUNIOR FJA, BONETTI FILHO I, SOUSA NETO MD, MIRANDA CES, J Appl Oral Sci, 2012;**20**(4):455-61

14.ICEANU MS, MELIAN A, HAMBURDA T, AMINOV L, VATAMAN M, Romanian Journal of Oral Rehabilitation, Vol. 5, No. 1, January - March 2013

15.KALASKAR R, TIKU A, DAMLE SG, J Indian SocPedPrev Dent. 2004;**22**:158-61

16.JOHNSON BR, WITHERSPOON DE, Periradicular Surgery. In Cohen S, Hargreaves KM, Pathways of the Pulp, 9th ed. Missouri: Mosby, 2006. pp. 724–85

17.VERGHESE GM, GEORGE T, KUTTAPPA MA, GIRISH K, Endodontology. 2009, 84–89

18.AL-HADDAD A, CHE AB AZIZ ZA. International Journal of Biomaterials. 2016:9753210. doi:10.1155/2016/9753210

19.NILSSON E, BONTE E, BAYET F, LASFARGUES JJ, International Journal of Dentistry, vol. 2013, Article ID 929486, 7 pages, 2013. doi:10.1155/2013/929486

20.SRIVASTAVA S, MAJUMDAR A, KOCHHAR R, DEWAN R, DHINGRA A, Endodontology, Vol. **26**, Issue 2, December 2014, pp. 305-308

21.MOHAMMADI Z, SHALAVI S, YAZDIZADEH M, Chonnam Medical Journal, 2012;**48**(3):133-140

22.SUZUKI P, de SOUZA V, HOLLAND R, et al. Journal of Applied Oral Science. 2011;19(5):511-516

23.ELDENIZ AU, ERDEMIR A, KURTOGLU F, ESENER T, Oral Surg Oral Med Oral Pathol Oral Radiol Endod. 2007;**103**(3):86-91

24.DUARTE MA, DEMARCHI AC, GIAXA MH, KUGA 3.MC, FRAGA SC, DE SOUZA LC, J Endod. 2000;26(7):389-9

25.VIVAN RR, ZAPATA RO, ZEFERINO MA, et al, Oral Surg Oral Med Oral Pathol Oral Radiol Endod. 2010;**110**(2):250-6

26.MASSI S, TANOMARU-FILHO M, SILVA GF, DUARTE MA, GRIZZO LT, BUZALAF MA, et al, J Endod. 2011;**37**(6):844-6

27.SIQUEIRA JF JR, DE UZEDA M, J Endod. 1996;22(12):674-6

28.CHÁVEZ-ANDRADE GM, KUGA MC, HUNGARO DUARTE MA, DE TOLEDO LEONARDO R, KEINE KC, SANT´ANNA-JR A, REIS SÓ MV, The Journal of Contemporary Dental Practice, November-December 2013;**14**(6):1094-1099

29.SHETTY V, HEGDE P, CHAUHAN RS, CHAURASIA VR, SHARMA AM, TARANATH M, Journal of International Oral Health/ : JIOH. 2015;7(2):25-27

30.SAUNDERS EM. Hand Instrumentation in root canal preparation. Endodontic Topics. 2005;**10**:163–7

31.KANDASWAMY D, VENKATESHBABU N, Journal of Conservative Dentistry/: JCD. 2010;13(4):256-264

32.ANSARI I, MARIA R, Contemporary Clinical Dentistry. 2012;3(2):237-241

33.JAVIDI M, ZAREI M, AFKHAMI F, MAJDI LMA, European Journal of Dentistry. 2013;7(1):69-73

34.ESTRELA C, HOLLAND R, ESTRELA CRA, Alencar AH G et al, Brazilian Dental Journal, 2014, **25**(1), 3-11

Manuscript received: 28.05.2017