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The Effect of Altered pH on Push-Out Bond Strength of Biodentin, Glass Ionomer Cement, Mineral Trioxide Aggregate and Theracal

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SUMMARY

Introduction Throughout the history of dentistry, a wide variety of materials such as gold-foil, silver posts, amalgam, zinc oxide eugenol, glass ionomer cements, mineral trioxide aggregate have been used as retrograde fillings. Altered pH in periapical lesions can affect push-out bond strength of these materials. The aim of this study was to evaluate the effect of altered pH on push-out bond strength of Biodentin, Glass ionomer cement (GIC), Mineral trioxide aggregate (MTA) and Theracal.

Material and Methods Forty-eight dentin slices of extracted single–rooted human teeth were sectioned and their canal portion instrumented to achieve a diameter of 1.4 mm. The specimens were then assigned into the four groups (one group for each material) with 12 samples in each group. All groups were further divided into 3 subgroups (with 4 specimens in each subgroup): acidic (butyric acid buffered at pH 6.4), neutral (phosphate buffer saline solution at pH 7.4) and alkaline (buffered potassium hydroxide at pH 8.4). Samples were incubated for 4 days at 37°C in acidic, neutral or alkaline medium. Push-out bond strength was measured using a Universal Testing Machine. The slices were examined under a stereomicroscope to determine the nature of bond failure.

Results GIC showed the highest bond strength (33.33MPa) in neutral and acidic medium (26.75MPa) compared to other materials. Biodentin showed the best result in alkaline medium.

Conclusion Altered pH level affected push-out bond strength of root end materials. GIC demonstrated good push-out bond strength that increased with decrease of pH whereas newer materials Biodentin and Theracal showed satisfying results in altered pH.

Keywords: push-out bond strength; altered pH; root end filling materials

INTRODUCTION

Infected or inflamed tissue may have a normal pH of 7.4 or an acidic pH as low as 5.0. Acidic pH may inhibit setting reaction, affect adhesion, or increase solubility of materials. If infection or inflammation persists, erosion of filling materials can occur in acidic environment generated by bacteria or inflammation [1]. Also, exposure of root end filling material to an alkaline environment after pretreatment with calcium hydroxide might affect its properties [2]. Therefore, sealing ability of material may be directly or indirectly affected by environmental conditions and pH of medium [1]. Over the years various root end filling materials such as gold-foil, silver posts, amalgam (with and without bonding agent), zinc oxide eugenol, glass ionomer cements, mineral trioxide aggregate have been used as retrograde fillings [3].

Glass ionomer cements (GICs) are widely used for a variety of purposes such as intermediate restorations, permanent restoration of micro-cavities, fissure sealing of erupting molars and root end filling material [4]. GICs are formed by the reaction between calcium–aluminosilicate glass particles and aqueous solutions of polyacrylic acid. The main advantages of GICs are their strong chemical bond with dentin and their ability to release fluoride ions. Due to this chemical bonding to dentin, GICs have shown higher bond strength even when used as root end filling material. It has been reported that these cements are easy to handle and they do not cause any adverse histological reaction in the periapical tissue [3]. However, push-out bond strength of GICs under altered pH has not been studied yet.

In the recent decades, mineral trioxide aggregate (MTA) has shown promising results when used as repair material of lateral root walls or furcation perforations, root-end filling, apical plug, and root canal filling [5, 6]. It consists of a fine powder of tricalcium silicate, dicalcium silicate, tricalcium aluminate, tetracalcium aluminoferrite, and bismuth oxide. During clinical application as root-end and perforation filling material or as an apical plug in necrotic teeth with open apices, MTA may be exposed to an acidic environment because of the presence of periradicular inflammation [7, 8]. pH change of host tissues because of the presence of pre-existing disease may affect physical and chemical properties of material [9, 10]. It has been reported that hardness [11], diametric tensile strength

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[8], push-out bond strength [10], and sealing ability of MTA [12] decrease after placement in acidic environment. As MTA has been shown to be suitable material, other calcium silicate–based materials have been developed recently to improve MTA drawbacks such as prolonged setting time, difficult handling, high cost, and potential tooth discoloration.

Recently, a new calcium silicate–based material Biodentin (Septodont, Saint-Maur-des-Fossés, France) has been introduced to the market. Biodentin is composed of tricalcium silicate, calcium carbonate, zirconium oxide, and a water-based liquid containing calcium chloride used as setting accelerator and water-reducing agent [13]. Biodentin is a fast-setting calcium silicate–based material that can be used as dentin restorative material as well as endodontic material with characteristics comparable to MTA [14].

Theracal (Bisco Inc, Schamburg, IL, USA) is a new light-cured resin-modified calcium silicate-filled base/liner material designed for direct and indirect pulp capping. It contains approximately 45% wt mineral material (type III Portland cement), 10% wt radiopaque component, 5% wt hydrophilic thickening agent (fumed silica) and approximately 45% resin [15]. It also shows physiochemical bonding to dentin, good sealing abilities and it is well tolerated by immortalized odontoblast cells [16, 17].

The aim of periradicular surgery is to remove the cause of disease and provide favourable environment for surgical wound healing. Placement of a root-end filling is one of the key steps in managing root end [18]. However, periapical lesions affect pH with consequent alteration of bond strength of root end filling materials.

The aim of the current study was to evaluate the effect of altered pH on push-out bond strength of Biodentin, Glass ionomer cement, Mineral trioxide aggregates and Theracal.

MATERIAL AND METHODS

Forty-eight single-rooted human anterior teeth with straight canals, extracted for periodontal and orthodontic reasons, were collected and stored in phosphate-buffered saline solution until used. Midroot dentin was sectioned horizontally into slices 1 mm thick. A diamond disc was used to obtain 48 root dentin slices (Figure 1). The canal portions of root dentin slices were instrumented to achieve a standardized diameter of 1.4 mm using round carbide bur (Figure 2). All used materials were mixed according to their manufacturers' instructions and introduced incrementally with no pressure into the lumens of the root-dentin slices. The specimens were then divided into the four groups (n=12), i.e. Biodentin, Glass ionomer cement, MTA (Mineral trioxide aggregates) and Theracal. These groups were further divided into 3 subgroups (n_s=4), i.e. acidic (butyric acid buffered at pH 6.4), neutral (phosphate buffer saline solution at pH 7.4) and alkaline (buffered potassium hydroxide at pH 8.4). They were then incubated for 4 days at 37°C.

Push-out bond strength was measured using a universal testing machine (Figure 3). The samples were placed

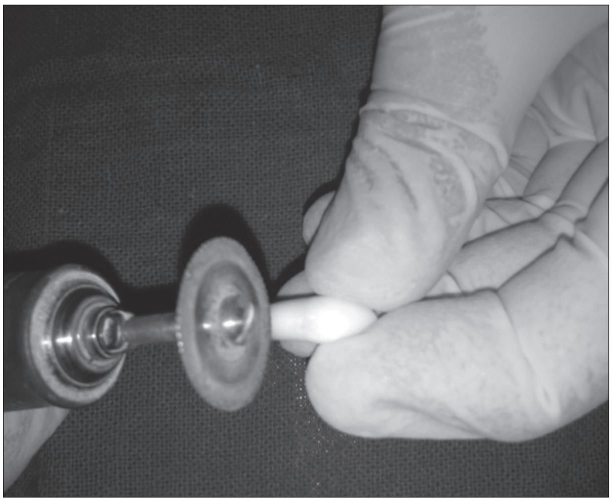


Figure 1. Mid-root dentin was sectioned horizontally using a diamond disc
Slika 1. Središnji deo korena zuba je horizontalno presečen dijamantskim diskom

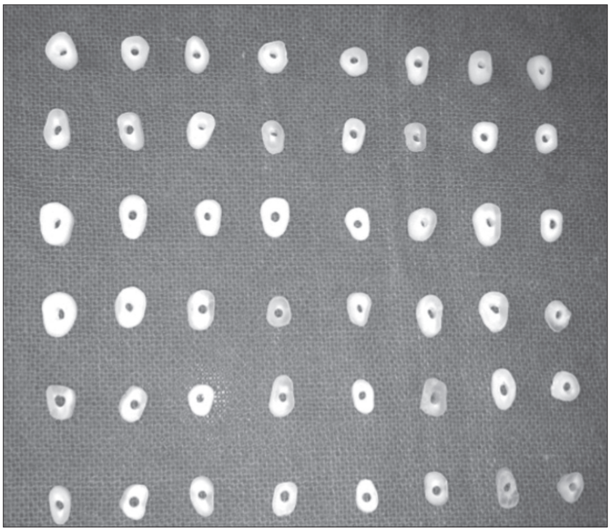


Figure 2. Forty-eight dentin slices were cut
Slika 2. Četrdeset osam dentinskih diskova je dobijeno

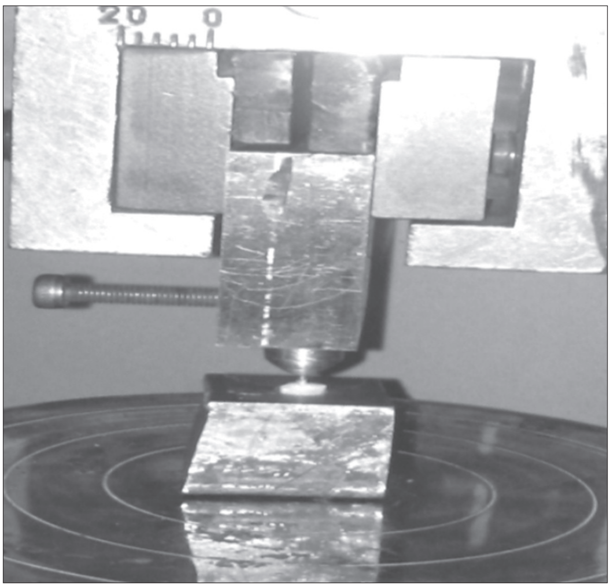


Figure 3. Universal testing machine
Slika 3. Univerzalna mašina za testiranje

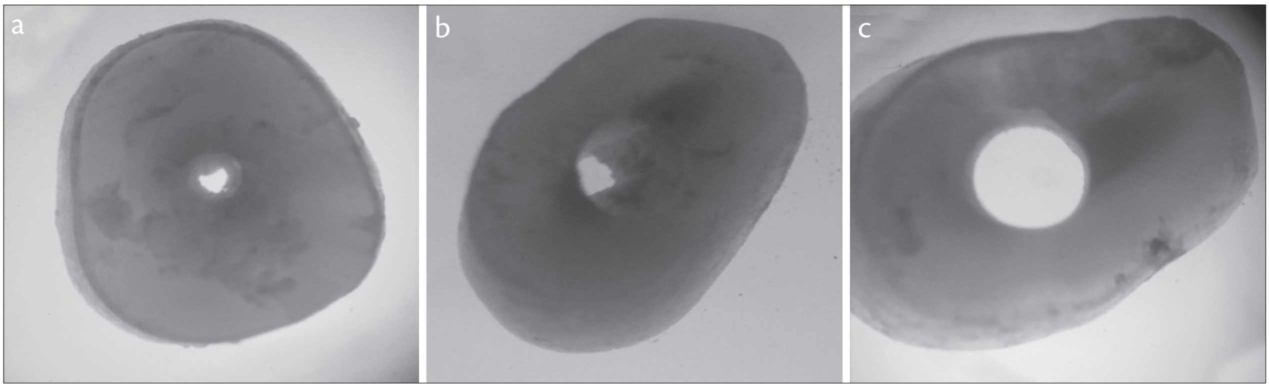


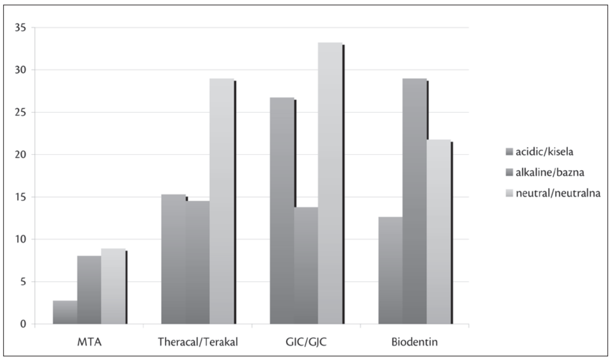
Figure 4. Bond failure: a) cohesive; b) mixed; c) adhesive
Slika 4. Oštećenje: a) kohezivno; b) mešovito; c) adhezivno

on a metal slab with a central hole to allow free motion of the plunger. Compressive load was applied by exerting downward pressure on the surface of materials using 1 mm diameter cylindrical jig at a speed of 1 mm/min. The jig had a clearance of approximately 0.2 mm from the margin of dentinal wall to insure contact with material only. Maximum load applied to material at the time of dislodgement was recorded in newton's.

In order to express bond strength in MPa, recorded value was divided by the adhesion area of root canal filling calculated by the following formula: $2\pi r \times h$, where r is the root canal radius and h is the thickness of the root-dentin slice in millimeters. The slices were then examined under a stereomicroscope to determine the nature of bond failure. Each sample was categorized into one of three failure modes: adhesive failure at the material and dentin interface, cohesive failure within materials, or mixed failure. The data were analyzed using one-way analysis of variance followed by the Tukey's Post Hoc Test.

RESULTS

Results showed a statistically significant difference among groups ($p<0.001$) after 4 days, where GIC had the highest bond strength in acidic and neutral environment while Biodentin showed the highest bond strength in alkaline environment. MTA showed the lowest push-out bond strength in all mediums compared to other root end filling materials. Statistical analysis also showed that the type of cement and storage solution significantly affected micro-push-out bond strength. Three modes of bond failure were found: adhesive failure at the material and dentin interface, cohesive failure within material, or mixed failure (Figure 4a-c; Graph 1; Table 1).



Graph 1. The effect of altered pH on push-out strength of different materials
Grafikon 1. Uticaj promenjene vrednosti pH sredine na otpornost na smicanje različitih materijala

Table 1. Type of bond failure in each group
Tabela 1. Tip oštećenja u svakoj grupi

Material + environment **Materijal + sredina**	**Type of bond failure** **Tip oštećenja**
GIC + acidic GJC + kisela	Cohesive Kohezivni
Biodentin + alkaline/neutral Biodentin + bazna/neutralna	
MTA + acidic/alkaline MTA + kisela/bazna	Adhesive Adhezivni
Theracal + acidic/alkaline/neutral Terakal + kisela/bazna/neutralna	
GIC + alkaline/neutral GJC + bazna/neutralna	
Biodentin + acidic BIodentin + kisela	Mixed Mešoviti
MTA + neutral MTA + neutralna	

Push-out bond strength:
In neutral medium: GIC (33.22 MPa) > Theracal (28.98 MPa) > Biodentin (21.76 MPa) > MTA (8.92 MPa). **In acidic medium:** GIC (26.75 MPa) >Theracal (15.29 MPa) > Biodentin (12.64 MPa) > MTA (2.78 MPa). **In alkaline medium:** Biodentin (28.89 MPa) > Theracal (14.54 MPa) > GIC (13.78 MPa) > MTA (8.03 MPa).

Otpornost na smicanje:
U neutralnoj sredini: GJC (33.22 MPa) > terakal (28.98 MPa) > biodentin (21.76 MPa) > MTA (8.92 MPa). **U kiseloj sredini:** GJC (26.75 MPa) > terakal (15.29 MPa) > biodentin (12.64 MPa) > MTA (2.78 MPa). **U baznoj sredini:** biodentin (28.89 MPa) > terakal (14.54 MPa) > GJC (13.78 MPa) > MTA (8.03 MPa).

DISCUSSION

Russian biologist Metchnikoff reported first evidence of acidic pH inside phagocytes in 1893. Later, Jensen and Bainton (1973) demonstrated that pH of a phagosome was reduced to approximately 6.5 within 3–4 min after initiation of phagocytosis. Also pH of pus aspirated from periapical tissues has been confirmed as acidic (6.68±0.324) [9]. Under certain clinical applications calcium silicate–based materials are used for the repair of root and furcation perforations, root-end fillings, and apical plugs. They are often placed in an environment where inflammation may be present and surface of unset material exposed to low pH. This altered pH may affect its physical and chemical properties [18, 19].

Normal tissue pH is 7.4 but it can be affected by certain clinical conditions. Tronstad et al. [20] showed pH in the range of 6.4–7 in the pulp, dentin, cementum, and periodontal ligament of vital or necrotic pulp teeth. As calcium hydroxide is preferred intracanal medicament, after its placement pH values of the most inner part of circumpulpal dentin change to pH range 11.1–12.2. It might be beneficial to provide pretreatment with calcium hydroxide in necrotic open apices or root perforations before application of biomaterials [21, 22]. There are conflicting results regarding the effect of calcium hydroxide dressing on sealing ability of various biomaterials [23, 24]. It has been suggested that residual calcium hydroxide might interfere with material adaptation to the root canal walls or chemically interact with them.

In the present study, push-out bond strength of Biodentin, Glass ionomer cement, MTA and Theracal was evaluated and compared after exposure to acidic, neutral and alkaline pH. For acidic environment, butyric acid, a byproduct of anaerobic bacteria metabolism, buffered at pH 6.4 was used. For alkaline environment buffered potassium hydroxide at pH 8.4 was used while for neutral environment phosphate buffer saline solution at pH 7.4 was used.

The results of present study indicate that push-out bond strength of GIC (26.75 MPa) in acidic medium was significantly higher while MTA had the lowest (2.78 MPa) push-out bond strength compared to other materials. In alkaline medium, the highest push-out bond strength was shown by Biodentin (28.89 MPa) and the lowest by MTA (8.03 MPa). In neutral medium, the greatest push-out bond strength was shown by GIC (33.22 MPa) and the lowest by MTA (8.92 MPa). MTA showed inferior bond strength in all environmental conditions, which is in agreement with Shokouhinejad et al. [10]. It is possible that pH inhibits setting reaction, affects adhesion, or increases solubility of calcium silicate–based materials eventually affecting mechanical properties of material including surface microhardness.

In the current study, Biodentin showed greater bond strength in both acidic and alkaline medium than MTA. Bond strength of MTA was most likely affected by the alkaline pH of dentin [12]. Biodentin also showed superior bond strength in alkaline medium than in neutral conditions. GIC demonstrated the highest bond strength compared to other root end filling materials in acidic and neutral pH. This result is probably related to to its strong chemical bonding to dentin.

Theracal is pulp-capping material but due to its good sealing ability we used it as root end filling material. It showed higher bond strength than MTA (8.03), almost comparable to GIC (13.78) in alkaline environment and higher than Biodentin in acidic environment. Further *in vivo* and *in vitro* research should be done in order to assess Theracal as root end filling material.

CONCLUSION

Altered pH levels affect the properties of root end materials. MTA showed inferior push-out bond strength that was affected by pH of surrounding environment. GIC has good bond strength that increased with decrease in pH opposite to other materials. Further research is needed for Biodentin and Theracal as root end filling materials.

REFERENCES

1. Roy CO, Jeansonne BG, Gerrets TF. Effect of an acid environment on leakage of root-end filling materials. J Endod. 2001; 27:7-8.
2. Saghiri MA, Shokouhinejad N, Lotfi M, Aminsobhani M, Saghiri AM. Push-out bond strength of mineral trioxide aggregate in the presence of alkaline pH. J Endod. 2010; 36:1856-9.
3. Vasudev SK, Goel BR, Tyagi S. Root end filling materials – a review. Endodontology. 2003; 15:12-8.
4. Zalizniak I, Palamara JE, Wong RH, Cochrane NJ, Burrow MF, Reynolds EC. Ion release and physical properties of CPP–ACP modified GIC in acid solutions. J Endod. 2013; 41:449-54.
5. Torabinejad M, Chivian N. Clinical applications of mineral trioxide aggregate. J Endod. 1999; 25:197-205.
6. Felippe WT, Felippe MC, Rocha MJ. The effect of mineral trioxide aggregate on the apexification and periapical healing of teeth with incomplete root formation. Int Endod J. 2006; 39:2-9.
7. Giuliani V, Nieri M, Pace R, Pagavino G. Effects of pH on surface hardness and microstructure of mineral trioxide aggregate and aureoseal: an in vitro study. J Endod. 2010; 36:1883-6.
8. Shie MY, Huang TH, Kao CT, Huang CH, Ding SJ. The effect of a physiologic solution pH on properties of white mineral trioxide aggregate. J Endod. 2009; 35:98-101.
9. Nekoofar MH, Namazikhah MS, Sheykhrezae MS, Mohammadi MM, Kazemi A, Aseeley Z, et al. pH of pus collected from periapical abscesses. Int Endod J. 2009; 42:534-8.
10. Shokouhinejad N, Nekoofar MH, Iravani A, Kharrazifard MJ, Dummer PM. Effect of acidic environment on the push-out bond strength of mineral trioxide aggregate. J Endod. 2010; 36:871-4.
11. Namazikhah MS, Nekoofar MH, Sheykhrezae MS, Salariyeh S, Hayes SJ, Bryant ST, et al. The effect of pH on surface hardness and microstructure of mineral trioxide aggregate. Int Endod J. 2008; 41:108-16.
12. Saghiri MA, Lotfi M, Saghiri AM, Vosoughhosseini S, Fatemi A, Shiezadeh V, et al. Effect of pH on sealing ability of white mineral trioxide aggregate as a root-end filling material. J Endod. 2008; 34:1226-9.
13. Han L, Okiji T. Uptake of calcium and silicon released from calcium silicate-based endodontic materials into root canal dentine. Int Endod J. 2011; 44:1081-7.
14. Laurent P, Camps J, De Meo M, Déjou J, About I. Induction of specific cell responses to a Ca(3)- SiO(5)-based posterior restorative material. Dent Mater. 2008; 24:1486-94.
15. Suh B, Cannon M, Yin R, Martin D. Polymerizable dental pulp healing, capping, and lining material and method for use. International Patent A61K33/42; A61K33/42 Application number WO2008US54387 20080220; Publication number WO2008103712 (A2); Publication date 2008-08-28.
16. Hebling J, Lessa FCR, Nogueira I, Carvalho RM, de Souza Costa CA. Cytotoxicity of resin-based light-cures liner cements. Am J Dent. 2009; 22:137-42.
17. Gandolfi MG, Siboni F, Prati C. Chemical-physical properties of TheraCal, a novel light-curable MTA-like material for pulp capping. Int Endod J. 2012; 45:571-9.
18. Elnaghy AM. Influence of acidic environment on properties of biodentine and white mineral trioxide aggregate: a comparative study. J Endod. 2014; 40:953-7.
19. Lee YL, Lee BS, Lin FH, Lin YA, Lan WH, Lin CP. Effects of physiological environments on the hydration behaviour of mineral trioxide aggregate. Biomaterials. 2004; 25:787-93.

20. Tronstad L, Andreasen JO, Hasselgren G, Kristerson L, Riis I. pH changes in dental tissues after root canal filling with calcium hydroxide. J Endod. 1981; 7:17-21.
21. Yildirim G, Dalci K. Treatment of lateral root perforation with mineral trioxide aggregate: a case report. Oral Surg Oral Med Oral Pathol Oral Radiol Endod. 2006; 102:e55-8.
22. Young GR. Contemporary management of lateral root perforation diagnosed with the aid of dental computed tomography. Aust Endod J. 2007; 33:112-8.
23. Hachmeister DR, Schindler WG, Walker WA 3rd, Thomas DD. The sealing ability and retention characteristics of mineral trioxide aggregate in a model of apexification. J Endod. 2002; 28:386-90.
24. Stefopoulos S, Tsatsas DV, Kerezoudis NP, Eliades G. Comparative in vitro study of the sealing efficiency of white vs grey ProRoot mineral trioxide aggregate formulas as apical barriers. Dent Traumatol. 2008; 24:207-13.

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Uticaj vrednosti pH na otpornost na smicanje biodentina, glasjonomer-cementa, mineralnog trioksidnog agregata i terakala

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KRATAK SADRŽAJ

Uvod Gledano kroz istoriju stomatologije, širok spektar materijala kao što su zlatne folije, srebrni kočići, amalgam, cink-oksid eugenol, glasjonomer-cement i mineralni trioksidni agregat korišćeni su za retrogradno punjenje kanala korena zuba. Pokazano je da pH u predelu periapikalne lezije utiče na otpornost na smicanje ovih materijala. Cilj ovog istraživanja bio je da se utvrdi uticaj izmenjene vrednosti pH na otpornost na smicanje biodentina, glasjonomer-cementa (GJC), mineralnog trioksidnog agregata (MTA) i terakala.

Materijal i metode rada Četrdeset osam jednokorenih zuba je horizontalno presečeno na dve različite visine da bi se dobilo 48 cilindričnih uzoraka. Njihov lumen je obrađen kako bi se postigao prečnik od 1,4 mm. Uzorci su zatim svrstani u četiri grupe (jedna grupa za svaki materijal) od po 12 uzoraka. Svaka grupa je dodatno podeljena na tri podgrupe sa po četiri uzorka: kiseli pH (puferizovan butanskom kiselinom na vrednost pH od 6,4), neutralni pH (puferizovan fosfatnim puferom fiziološkog rastvora na pH od 7,4) i bazni pH (puferizovan kalijum-hidroksidom na pH od 8,4). Uzorci su inkubirani tokom četiri dana na 37°C u kiseloj, neutralnoj ili baznoj sredini. Otpornost na smicanje je merena pomoću univerzalne mašine za testiranje (engl. *Universal Testing Machine*). Uzorci su takođe posmatrani pod stereomikroskopom, kako bi se utvrdila priroda prekida veze materijala sa dentinom.

Rezultati Najveću otpornost na smicanje pokazao je GJC (33,33 MPa) u neutralnoj i kiseloj sredini (26,75 MPa) u poređenju sa drugim materijalima. Biodentin je pokazao najbolji rezultat u baznoj sredini.

Zaključak Izmenjena vrednost pH utiče na otpornost na smicanje materijala za punjenje apeksa kanala korena zuba. GJC je pokazao dobru otpornost na smicanje koja se poboljšavala sa smanjenjem vrednosti pH. Noviji materijali, biodentin i terakal, pokazali su zadovoljavajuće rezultate u izmenjenom pH sredine. MTA je pokazao najslabiju otpornost na smicanje.

Ključne reči: otpornost na smicanje; promena vrednosti pH; materijal za punjenje apeksa kanala korena zuba

UVOD

Inficirana i upaljena tkiva mogu imati normalnu vrednost pH od 7,4, ali i kiseli pH od oko 5,0. Kiseli pH može sprečiti uspostavljanje reakcije, uticati na prianjanje ili povećati rastvorljivost materijala. Ako infekcija ili upala traju dugo, to može dovesti i do erozije materijala [1]. S druge strane, i izloženost baznoj sredini nastaloj nakon medikacije kanala kalcijum-hidroksidom može uticati na svojstva materijala za punjenje apeksa korena zuba [2]. Na taj način sposobnost opturacije materijala može biti direktno ili indirektno poremećena [1]. Kroz istoriju stomatologije različiti materijali su korišćeni za retrogradno punjenje kanala korena, kao što su zlatne folije, srebrni kočići, amalgam (sa adhezivom i bez njega), cink-oksidni eugenol, glasjonomer-cement, mineralni trioksidni agregat i dr. [3].

Glasjonomer-cement (GJC) se koristi u različite svrhe, kao što su privremeni ispuni, stalni ispuni malih kaviteta, zalivanje fisura i punjenje apeksa korena [4]. GJC se formira reakcijom čestica kalcijum-aluminosilikatnog stakla i vodenog rastvora poliakrilne kiseline. Glavna prednost ovog cementa je jaka hemijska veza sa dentinom i sposobnost oslobađanja jona fluorida. Zahvaljujući dobroj hemijskoj vezi sa dentinom, GJC pokazuje dobru vezu kada se koristi kao materijal za punjenje apeksa korena. Ovaj cement je jednostavan za upotrebu i ne dovodi do loših reakcija na histološkom nivou u periapikalnom tkivu [3]. Dosada nije objavljena nijedna studija koja meri otpornost na smicanje GJC u uslovima izmenjene vrednosti pH.

U poslednjih nekoliko decenija mineralni trioksidni agregat (MTA) se pokazao vrlo uspešnim kada se koristi kao materijal za zatvaranje perforacija (bočnih zidova ili furkacija), kao apikalni čep i kao materijal za punjenje kanala korena [5, 6]. Sastoji se od finog praha trikalcijum-silikata, dikalcijum-silikata, trikalcijum-aluminata, tetrakalciujm-aluminoferita i bizmut-oksida. S obzirom na njegovu kliničku upotrebu, MTA može biti izložen kiseloj sredini zbog postojanja periapikalnog zapaljenja [7, 8]. Promena vrednosti pH zbog već postojećeg stanja može uticati na fizičke i hemijske osobine materijala [9, 10]. Tako je pokazano da se tvrdoća [11], dužinska zatezna čvrstoća [8], otpornost na smicanje [10] i sposobnost zaptivanja MTA [12] smanjuju nakon postavljanja materijala u kiseloj sredini. Novi materijali na bazi kalcijum-silikata uvedeni su da bi se prevazišli nedostaci MTA poput dužeg vremena vezivanja, teškog rukovanja, visoke cene i potencijalnog prebojavanja zuba.

Najnoviji na tržištu je kalcijum-silikatni materijal biodentin (*Septodont*, *Saint-Maur-des-Fossés*, Francuska), koji se sastoji od trikalcijum-silikata, kalcijum-karbonata, cirkonijum-oksida i tečnosti na bazi vode koja sadrži kalcijum-hlorid kao akcelerator i agens za oduzimanje vode [13]. Biodentin je brzovezujući kalcijum-silikatni materijal koji se može koristiti kao restorativni materijal za dentin, ali i kao endodontski materijal sa osobinama sličnim MTA [14].

Terakal (*Bisco Inc*, *Schamburg, IL*, SAD) je novi svetlosno-polimerizujući kalcijum-silikatni cement modifikovan smolom (baza/lajner) dizajniran za direktno i indirektno prekrivanje pulpe. On sadrži oko 45% mineralnih materijala po težini (tip III Portland cementa), 10% rendgenkontrastne komponente, 5% hidrofilnog agensa za zgušnjavanje (fumed silika) i oko 45% smole [15]. Pokazuje dobru fizičkohemijsku vezu za dentin, dobro zaptivanje i dobru toleranciju od strane odontoblasta [16, 17].

Cilj periapikalne hirurgije je da ukloni uzrok bolesti i omogući povoljno okruženje za zarastanje. Apikalno punjenje je jedno od ključnih koraka [18]. Međutim, postojanje periapikalne lezije utiče na promenu vrednosti pH, što dovodi do smanjenja kvaliteta veze s materijalom za punjenje apeksa kanala korena.

Cilj ovog rada je bio da se proceni efekat izmenjene vrednosti pH na otpornost na smicanje biodentina, GJC, MTA i terakala.

MATERIJAL I METODE RADA

U studiju je bilo uključeno 48 jednokorenih zuba s pravim kanalima koji su bili ekstrahovani iz parodontoloških ili ortodontskih razloga. Pre eksperimenta zubi su čuvani u puferizovanom fiziološkom rastvoru. Svaki koren zuba je presečen horizontalno dijamantskim diskom, da bi se dobili dentinski diskovi debljine 1 mm – ukupno 48 diskova (Slika 1). Deo kanala svakog diska je obrađen okruglim karbidnim svrdlom, da bi se dobio prečnik od 1,4 mm (Slika 2). Testirani materijali su zamešani prema uputstvu proizvođača i u inkrementima uneseni u lumen kanala svakog dentinskog diska. Uzorci su zatim svrstani u četiri grupe od po 12 uzoraka: biodentin, GJC, MTA i terakal. Potom je svaka grupa podeljena na tri podgrupe od po četiri uzorka: kisela sredina (butirična kiselina puferovana na vrednosti pH od 6,4), neutralna sredina (fiziološki rastvor puferizovan fosfatnim puferom na pH 7,4) i bazna sredina (puferizovana kalijum-hidroksidom na pH 8,4). Svi uzorci su inkubirani četiri dana na 37°C.

Otpornost na smicanje (tzv. *push-out* čvrstoća) svakog materijala merena je pomoću univerzalne mašine za merenje (Slika 3). Uzorci su postavljeni na metalnu ploču s otvorom u centru, da bi se klip mogao slobodno kretati. Pritisno opterećenje je aplikovano pritiskom nadole na površinu materijala pomoću cilindra prečnika 1 mm brzinom od 1 mm u minuti. Pritisni cilindar je bio bar 0,2 mm daleko od dentinskog zida, kako bi se osigurao kontakt samo s materijalom. Maksimalno opterećenje koje je podneo materijal u trenutku izbacivanja izražen je u njutnima (N).

Kako bi izrazili čvrstoću u MPa, zabeležena vrednost je podeljena površinom materijala u kanalu korena prema sledećoj formuli: $2\pi r \times h$; gdje je r prečnik kanala, a h debljina dentinskog diska izražena u mm. Svaki dentinski disk je potom ispitan pomoću stereomikroskopa, da bi se utvrdila priroda prekida kontakta između materijala i dentina, i to kao: narušavanje adhezivne veze između materijala i dentina, narušavanje kohezivne veze unutar materijala ili mešoviti prekid kontakta. Prikupljeni podaci su analizirani pomoću jednosmerne analize varijanse i *post hoc* Takijevim (*Tukey*) testom.

REZULTATI

Rezultati su pokazali statistički značajnu razliku između posmatranih grupa uzoraka nakon četiri dana ($p<0{,}001$), s tim da je GJC pokazao najveću čvrstoću u kiseloj i neutralnoj sredini, a biodentin u baznoj sredini. MTA je pokazao najmanju otpornost na smicanje u svim medijima u poređenju sa drugim materijalima. Statistička analiza je takođe pokazala da vrsta cementa i sredina značajno utiču na mikro *push-out* čvrstoću. Takođe su otkrivena sva tri načina kidanja veze između materijala i dentina, i to: adhezivni, kohezivni i mešoviti (Slika 4a-c; Grafikon 1; Tabela 1).

DISKUSIJA

Ruski biolog Mečnikov potvrdio je prvi put kiseli pH unutar fagocita 1893. godine. Kasnije su Jensen (*Jensen*) i Bejnton (*Bainton*) 1973. godine pokazali da pH u fagozomu dostiže oko 6,5 u roku od tri-četiri minuta nakon početka fagocitoze. Takođe je pokazano da je pH gnoja aspiriranog iz periapikalnih apscesa kiseo (6,68±0,324) [9]. Pod određenim kliničkim uslovima materijali na bazi kalcijum-silikata se koriste za opturaciju perforacije korena, apeksa kanala ili kao apikalni čepovi. Oni se često unose u sredinu u kojoj postoji upalni proces, tako da površina materijala može biti izložena niskoj vrednosti pH. Promenjena vrednost pH može uticati na fizička i hemijska svojstva materijala [18, 19].

Normalna vrednost pH zdravih tkiva je oko 7,4 i na njega mogu uticati različita klinička stanja. Tronštad (*Tronstad*) i saradnici [20] su pokazali da je pH pulpe, dentina, cementa i parodontalnog ligamenta vitalnih ili nekrotičnih zuba u rasponu od 6,4 do 7. Kako se kalcijum-hidroksid veoma često koristi kao intrakanalni medikament, nakon njegove upotrebe pH vrednost cirkumpulpalnog dentina dostiže vrednosti 11,1–12,2. Interseansna primena kalcijum-hidroksida je značajna u terapiji nekrotične pulpe kod otvorenih apeksa ili perforacija [21, 22]. U literaturi postoje oprečni rezultati koji se odnose na uticaj kalcijum-hidroksida na zaptivanje raznih biomaterijala [23, 24]. Zaostali kalcijum-hidroksid bi mogao fizički ometati adaptaciju materijala uz zidove korenova ili hemijski reagovati s njima.

U ovoj studiji otpornost na smicanje biodentina, GJC, MTA i terakala je procenjivana nakon izlaganja materijala kiselom, neutralnom i baznom pH. Za postizanje kisele sredine upotrebljena je butirična kiselina puferovana na vrednost pH od 6,4. Bazna sredina je postignuta rastvorom kalijum-hidroksida puferovanim na pH 8,4, dok je za neutralnu sredinu korišćen neutralni fosfatni pufer na pH 7,4.

Rezultati ovog istraživanja su pokazali da je otpornost na smicanje GJC u kiseloj sredini bila najveća (26,75 MPa), a MTA najniža (2,78 MPa). U baznoj sredini najveću otpornost na smicanje pokazao je biodentin (28,89 MPa), a najnižu MTA (8,03 MPa). U neutralnoj sredini najveću otpornost na smicanje opet je pokazao GJC (33,22 MPa), a najnižu MTA (8,92 MPa). MTA je pokazao slabiju čvrstoću u odnosu na druge materijale pri svim pH vrednostima, što je u skladu s nalazima Šokuhinedžada (*Shokouhinejad*) i saradnika [10]. Moguće je da pH blokira reakciju stvrdnjavanja, utiče na adheziju ili povećava rastvorljivost materijala na bazi kalcijum-silikata, što takođe utiče na mehanička svojstva materijala, uključujući i mikrotvrdoću.

U našoj studiji biodentin je pokazao veću čvrstoću i u kiseloj i u baznoj sredini nego MTA. Na jačinu veze MTA i dentina najverovatnije je uticao bazni pH dentina [12]. Biodentin je pokazao bolju čvrstoću u baznoj sredini nego u neutralnoj. GJC je pokazao najbolju čvrstoću od svih materijala u kiselom i neutralnom pH. Ovaj rezultat je verovatno posledica snažne hemijske veze sa dentinom.

Terakal je materijal za pokrivanje pulpe, a zbog svog dobrog rubnog zaptivanja izabran je za ovu studiju. On je pokazao veću čvrstoću nego MTA (8,03), gotovo uporedivu sa GJC (13,78), u baznoj sredini i veću nego biodentin u kiseloj sredini. Ipak, potrebna su dodatna istraživanja *in vivo* i *in vitro* da bi se terakal uspešno primenio kao materijal za punjenje apeksa kanala korena zuba.

ZAKLJUČAK

Promene vrednosti pH sredine utiču na svojstva materijala za punjenje apeksa korena. MTA je pokazao slabiju otpornost na smicanje, na koju je uticala promena pH sredine. GJC je pokazao dobru čvrstoću, koja se poboljšavala sa smanjenjem vrednosti pH sredine, što je bilo u suprotnosti sa drugim materijalima. Potrebna su dodatna istraživanja za procenu biodentina i terakala kao materijala za punjenje apeksa kanala korena.