Ultrastructural Analysis of the Surface of Endodontic Instruments after Immersion in Irrigating Solutions

Jelena Popović, Goran Radenković, Jovanka Gašić, Aleksandar Mitić, Marija Nikolić, Radomir Barac, Slavoljub Živković

1University of Niš, Medical Faculty, Clinic of Dentistry, Department of Restorative Dentistry and Endodontics, Niš, Serbia; 2University of Niš, Mechanical Faculty, Department of Production Engineering, Niš, Serbia; 3University of Belgrade, Faculty of Dental Medicine, Department of Restorative Dentistry and Endodontics, Belgrade, Serbia

SUMMARY

Introduction Separation (fracture) of endodontic instruments in the root canal during chemomechanical instrumentation is a complication that can compromise the final outcome of endodontic treatment. One of the most common factors that cause fatigue of endodontic instruments and consequent fracture is surface corrosion. The aim of this study was to investigate the ultrastructure of surface corrosion of endodontic instruments made of stainless steel and nickel-titanium after immersion in the most commonly used root canal irrigants.

Material and Methods The study included 48 nickel-titanium and stainless steel endodontic hand files. All instruments were immersed in 5.25% sodium hypochlorite, 0.2% CHX and 17% EDTA. Surface corrosion was analyzed using a scanning electron microscope (SEM).

Results Nickel-titanium instruments showed significantly higher susceptibility to corrosion after immersion in 5.25% sodium hypochlorite compared to stainless steel instruments (p<0.001). After immersion in 0.2% CHX corrosion damage was observed on both nickel-titanium and stainless steel instruments but the difference was not statistically significant (p=0.096). No corrosion was observed in both types of instruments after immersion in 17% EDTA.

Conclusion The use of 5.25% NaOCl and 0.2% CHX as root canal irrigating solutions can cause serious corrosion changes on the surface of nickel-titanium and stainless steel endodontic instruments.

Keywords: corrosion; endodontic instruments; nickel-titanium; stainless steel; SEM

INTRODUCTION

Endodontic instrument separation (fracture) in the root canal during chemomechanical preparation is a complication that can compromise the final outcome of endodontic therapy [1]. Weakening of instruments’ structure is one of important factors that affect safety of their use. Numerous studies that have examined clinical use of endodontic instruments have concluded that metal fatigue is the first anomaly which occurs during clinical use and that, combined with sudden loads during root canal instrumentation, may lead to fracture [1, 2]. In addition, the literature data indicate surface corrosion of endodontic instruments as one of the first factors that can cause fatigue of the material [3]. Corrosion can start during chemomechanical instrumentation or chemical disinfection and sterilization of instruments [1]. Corrosion is caused by the contact of metal with different solutions when various electrochemical reactions occur and affect surface integrity making instruments more prone to fracture [4]. Corrosion pits and surface porosity can also reduce the cutting efficiency of endodontic instruments [5].

The aim of this study was to evaluate the ultrastructure of surface corrosion of stainless steel and nickel-titanium endodontic files after immersion in the most commonly used root canal irrigants.

MATERIAL AND METHODS

The study included nickel-titanium (Ni-Ti) („I-FLEX“, „IMD“, USA) and stainless steel (NTI-Kahla GmbH, Germany) hand endodontic files. New instruments were taken out of the packages, and in order to remove all debris received from manufacturers, the files were cleaned in the ultrasonic bath (JUS-S01, JEOL) with distilled water for 15 minutes at the frequency of 28kHz. Then after, the corrosion behaviour of endodontic instruments was assessed using potentiodynamic method in the three most commonly used irrigating solutions: 5.25% sodium hypochlorite (NaOCl) (prepared in the laboratory); 0.2% chlorhexidine gluconate (CHX) (R4, Septodont, France, diluted to 0.2%); 17% ethylenediamine tetraacetic acid (EDTA) (prepared in the laboratory).
All solutions used in this study were freshly prepared and stored in adequate conditions. Forty-eight instruments (24 Ni-Ti and 24 stainless steel instruments) were tested. Instruments were divided into the 6 groups according to the material and used irrigating solution (Table 1).

The experiments were carried out in an ordinary, three-compartment cylindrical glass cell. The counter electrode was a Pt foil and the reference electrode was a saturated calomel electrode (SCE). All potentials were referred to SCE. The working electrode - endodontic instrument was placed into the cell in such way that only working part of the instrument was immersed in the solution, whereas the handle was above the solution. The instruments were immersed 15 seconds before the potential rise as set by the software. Anodic E-I polarization curves were recorded using the software Par Stat by means of the linear sweep technique (sweep rate 0.2mV/s). The potential value that showed sharp rise of the current was assigned as pitting potential. The sharp increase of the current was a result of local dissolution of metal and forming pits. Electrochemical testing was performed at the Department of Production Engineering, Faculty of Mechanical Engineering, University of Niš, and Department of Physical Chemistry and Electrochemistry, Faculty of Technology and Metallurgy, University of Belgrade.

After electrochemical testing (published results) [6], the instruments were prepared for scanning electron microscopic examination (SEM; JEOL-JSM 5300). In order to obtain adequate visualization of the working parts of instruments, the handles of the instruments were cut off, and their working parts were fixed to the aluminum stubs with a fixing agent (Dotite paint xc 12 Carbon JEOL, Tokyo, Japan) and sputter coated with gold/palladium (in the unit JFC 110 Ion Sputter JEOL). SEM examination was completed at the Institute for Biomedical Research of the Faculty of Medicine in Nis.

Ultrastructural analysis of the surface corrosion changes was analysed using modified score presented by Linsuwanont et al. [7]: score 3 – continuous corrosion of the entire surface of the instrument; score 2 – clearly limited corrosion fields; score 1 – individual corrosion pits; score 0 – no visible corrosion changes. Surface of the working parts of the instruments was observed at three levels: apical, middle and coronal. At each level an appropriate score was estimated, and final score represented the mean value of all three scores for each instrument individually and also within the groups.

Statistical analysis was performed using χ² and Fisher Exact test, a p value of p<0.05 was considered statistically significant.

### RESULTS

Ultrastructural analysis of instruments surfaces showed the most intensive corrosion changes on the Ni-Ti instruments after immersion in 5.25% sodium hypochlorite. Erosive metal surfaces were observed along the entire working part of all tested instruments in this group (score 3) (Table 2, Figures 1 and 2). Sensitivity to 5.25% NaOCl was also seen in stainless steel instruments. Continuous surface corrosion and limited fields of corrosion were observed on the working surfaces of these instruments, so the total score of this group was 2.33 (Table 2, Figure 3). Fisher Exact test showed significantly higher sensitivity of Ni-Ti instruments compared to stainless steel after immersion in 5.25% sodium hypochlorite (p<0.001).

SEM analysis of the Ni-Ti instruments after immersion in 0.2% CHX showed limited corrosion fields and individual corrosion pits. The total score in this group of instruments was 1.5 (Table 2, Figures 4 and 5). Stainless steel instruments showed higher vulnerability to 0.2% CHX. These instruments had higher number of limited corrosion fields compared to individual corrosion pits, so the total score in this group was 1.83 (Table 2, Figure 6). However, Fisher Exact test did not show statistically significant difference in sensitivity to 0.2% CHX between both types of instruments (p=0.096).

Ultrastructural analysis of Ni-Ti instruments after immersion in 17% EDTA did not show corrosion defects on the surface of working parts of the instruments so this group was assigned score 0 (Table 2, Figure 7). Corrosion damages were not observed on the surfaces of the stainless steel instruments after immersion in 17% EDTA, so the average score for this group was also 0 (Table 2, Figure 8).

Nickel-titanium and stainless steel instruments showed different susceptibility to the tested irrigating solutions. Nickel-titanium instruments showed significantly higher susceptibility to 5.25% sodium hypochlorite compared to

### Table 1. Endodontic instruments according to the material and irrigating solution

| Material Medijal | Irrigating solution Rastvor za irrigaciju | Control Kontrola | Total Ukupno |
|------------------|------------------------------------------|-----------------|--------------|
| Ni-Ti            | NaOCl 6 CHX 6 EDTA 6                  | 6               | 24           |
| Stainless steel | NaOCl 6 CHX 6 EDTA 6                  | 6               | 24           |
| Total            | 12 12 12 12                            | 12              | 48           |

Ni-Ti – nickel-titanium; NaOCl – sodium hypochlorite (5.25%); CHX – chlorhexidine gluconate (0.2%); EDTA – ethylenediamine tetraacetic acid (17%)
Ni-Ti – nikel-titanijum; NaOCl – natrijum-hipohlorit (5,25%;) CHX – hlorheksidin-glukonat (0,2%); EDTA – etilendiaminotetrasirćetna kiselina (17%)

### Table 2. Average score of corrosion defects on instruments after immersion in endodontic irrigants

| Material Medijal | Irrigating solution Rastvor za irrigaciju | Control Kontrola |
|------------------|------------------------------------------|-----------------|
| Ni-Ti            | NaOCl 3 CHX 1.5 EDTA 0                  | 0               |
| Stainless steel | NaOCl 2.33 CHX 1.83 EDTA 0             | 0               |

Ukupno 12 12 12 12 48
Total
Stainless steel
Table 1.
Podела испитаних инструмената у односу на врсту материјала и раствор за иригацију

Tabela 1.
Podela ispitivanih instrumenata u odnosu na vrstu materijala i rastvor za irrigaciju

Tabela 2. Prosečan skor korozivnih oštećenja na instrumentima posle potapanja u rastvore za irigaciju
Figure 1. Continuous corrosion damage of the nickel-titanium (Ni-Ti) instrument surface after immersion in 5.25% sodium hypochlorite (NaOCl).  
Slika 1. Kontinuirano korozivno oštećenje površine instrumenta od nikl-titanijuma (Ni-Ti) posle potapanja u rastvor natrijum-hipohlorita (NaOCl) u koncentraciji od 5.25%.

Figure 2. Surface erosion of the Ni-Ti instruments after immersion in 5.25% NaOCl at ×500 magnification.  
Slika 2. Površinska erozija Ni-Ti instrumenta posle potapanja u 5,25% NaOCl pri uvećenju od ×500.

Figure 3. Continuous surface corrosion of the stainless steel instrument after immersion in 5.25% NaOCl.  
Slika 3. Kontinuirana korozivna površina instrumenta od nerđajućeg čelika posle potapanja u 5,25% NaOCl.

Figure 4. Limited corrosion fields and individual corrosion pits on the surface of the Ni-Ti instrument after immersion in 0.2% chlorhexidine gluconate (CHX).  
Slika 4. Ograničena korozivna polja i pojedinačne korozivne jamice na površini Ni-Ti instrumenta posle potapanja u rastvor hlorheksidin-glukonata (CHX) u koncentraciji od 0,2%.

Figure 5. Limited corrosion fields and individual corrosion pits on the surface of the Ni-Ti instrument after immersion in 0.2% CHX at higher magnification.  
Slika 5. Ograničena korozivna polja i pojedinačne korozivne jamice na površini Ni-Ti instrumenta posle potapanja u 0,2% CHX na većem uvećanju.

Figure 6. Corrosion in the form of restricted fields on the stainless steel instrument after immersion in 0.2% CHX.  
Slika 6. Korozija u vidu ograničenih polja na instrumentu od nerđajućeg čelika posle potapanja u 0,2% CHX.
0.2% CHX, $\chi^2$ test showed that this difference was highly statistically significant ($p<0.001$). Stainless steel instruments also showed statistically higher sensitivity to immersion in 5.25% sodium hypochlorite compared to 0.2% CHX ($p<0.05$).

**DISCUSSION**

Chemomechanical instrumentation of the root canal is essential during endodontic treatment and involves procedures of cleaning and shaping of the root canal space and use of irrigating solutions. Most commonly used irrigation solutions are: sodium hypochlorite (NaOCl), hydrogen peroxide (H$_2$O$_2$), citric acid, ethylenediaminetetraacetic acid (EDTA), chlorhexidine gluconate (CHX), saline solution, etc [5]. Although the use of irrigants during root canal preparation is essential, chemical and electrochemical aggressiveness of these solutions can damage surface of the instruments [8].

Electrochemical techniques based on determination of pitting potential and current density can accurately define sensitivity of metals to different solutions [9]. The surface ultrastructure also plays an important role in determining corrosion behaviour of the tested endodontic instruments in certain solutions [10]. Corrosion on the microscopic level is directly related to the weakening of the structure of instruments that reduces cutting efficiency and make instruments more susceptible to fracture [9].

Corrosion behaviour of nickel-titanium and stainless steel instruments can also increase the severity of surface attack and dissolution of metal surface [9]. Sodium hypochlorite (NaOCl) is the most commonly used solution for root canal irrigation in endodontic practice. It is used in concentration range from 0.5% to 6% [12]. It has a wide spectrum of antimicrobial activity, and due to its ability to dissolve organic part of dentin it is used for removing smear layer as well as pre-soaking solution in the cleaning procedures after clinical use [13, 14]. However, sodium hypochlorite contains active and aggressive Cl$^-$ ions that promote the occurrence of corrosion pits and weakening of the instrument structure [15]. It has been shown that NaOCl is corrosive for many metals and selectively removes nickel from Ni-Ti alloys [16]. Studies have shown measurable release of titanium after immersion of Ni-Ti instruments in NaOCl for 30 to 60 minutes [17]. Sensitivity of nickel-titanium and stainless steel endodontic instruments to NaOCl has been reported in numerous studies [1, 5, 13]. In the study of Stokes et al. [14] corrosion of endodontic instruments was visually confirmed after immersion in 5.25% NaOCl. A significant difference between different manufacturers was observed, but there were no significant differences between nickel-titanium and stainless steel instruments. Berutti et al. [4] found that instruments immersed in NaOCl had significantly reduced resistance to fracture due to early cycle fatigue and occurrence of unexpected fractures in these instruments was significantly higher than in the control group of instruments. SEM analysis of the fractured surface revealed limited corrosion fields, pits and cracks. The effect of Cl$^-$ and F$^-$ ions on the corrosion of Ni-Ti and stainless steel was studied by Amaral et al. [18] and Aboud et al. [19] for the purpose of electrochemical dissolution and removal of fractured endodontic instruments from root canals. The current study revealed intensive continuous corrosion damages of instruments after immersion in 5.25% sodium hypochlorite and this was in accordance with pitting potential values obtained in electrochemical analysis [6].
Chlorhexidine gluconate (CHX) represent frequently used root canal irrigant due to its prolonged antimicrobial effect that may last up to 12 weeks [20]. It is used in a concentration range from 0.1% to 2%. However, literature data indicate the potential for surface corrosion of instruments after immersion in CHX [5, 6]. Corrosion potential of CHX depends on its acidic pH (5.72) as acidic environment increases the corrosion rate [21]. In the current study, a visibly damage of the surface of Ni-Ti and stainless steel instruments was observed after immersion in 0.2% CHX. Such visible damage in the form of limited fields and fissures can act as weak points where further loads on instruments can lead to undesirable cracks that propagate [8].

Ethylenediamine tetraacetic acid (EDTA) is a chelating agent that is used in endodontic practice at concentrations from 15% to 17%. Due to its ability to dissolve inorganic part of dentin, it is used as a lubricant in the preparation of narrow and curved root canals and for removal of inorganic part of the smear layer [22]. The results of SEM analysis from the current study revealed no negative effects of EDTA on the surface structure of Ni-Ti and stainless steel instruments, and that was in accordance with the results of electrochemical testing from previous study [6]. In a study published by Fayyad and Mahran [23] there was no visible change in surface roughness of endodontic instruments after immersion in 17% EDTA. According to Reinhard et al. [24] EDTA has the ability to protect and passivize instruments because it forms complexes with metal ions at pH values less than 4 thus creating an inhibiting barrier for oxidation and corrosion.

CONCLUSION

The use of 5.25% NaOCl and 0.2% CHX as root canal irrigants may cause serious corrosion damage on the surface of Ni-Ti and stainless steel endodontic instruments. The application of 17% EDTA did not cause corrosion changes in both types of instruments. To minimize the risk of damage it is recommended that irrigants should be rinsed out from the files immediately after their use and files should be replaced frequently.

NOTE

The paper was given as poster presentation at the Rosov Pin 2014, The Second Regional Roundtable: Refractory, Process Industry and Nanotechnology, held on October 23-24, 2014 in Fruška gora.

REFERENCES

1. Darabara M, Bourithis L, Zinelis S, Papadimitriou GD. Susceptibility to localized corrosion of stainless steel and NiTi endodontic instruments in irrigating solutions. Int Endod J. 2004; 37:705-10. [DOI: 10.1111/j.1365-2591.2004.00866.x] [PMID: 15347296]

2. Zinelis S, Margelos J. Failure mechanism of Hedstrom endodontic files in vivo. J Endod. 2002; 28:471-3. [DOI: 10.1097/00004770-200206000-00014] [PMID: 12067133]

3. Sotokawa T. An analysis of clinical breakage of root canal instruments. J Endod. 1988; 14:75-82. [DOI: 10.1016/S0099-2399(88)80005-0] [PMID: 3162943]

4. Berutti E, Angelini E, Rigolone M, Migliaretti G, Pasqualini D. Influence of sodium hypochlorite on fracture properties and corrosion of ProTaper rotary instruments. Int Endod J. 2006; 39:693-9. [DOI: 10.1111/j.1365-2918.2006.00134.x] [PMID: 16916358]

5. Öztan DM, Akman AA, Zaimoglu L, Bilgić S. Corrosion rates of stainless-steel files in different irrigating solutions. Int Endod J. 2002; 35:655-9. [DOI: 10.1111/j.1365-2918.2002.00530.x] [PMID: 12196218]

6. Popović J, Radenković Ć, Gašić J, Živković S, Mitić A, Nikolić M, et al. The examination of sensitivity to corrosion of nickel-titanium and stainless steel endodontic instruments in root canal irrigating solutions. Chemical Industry and Chemical Engineering Quarterly. [in press] [DOI: 10.2298/CICEQ1503033PP]

7. Linsuwanont P, Parashos P, Messer HH. Cleaning of rotary nickel-titanium endodontic instruments. Int Endod J. 2004; 37:19-28. [DOI: 10.1111/j.1365-2918.2004.00747.x] [PMID: 14718053]

8. Saglam BC, Kocak S, Kocak MM, Topuz O. Effects of irrigating solutions on the surface of ProTaper instruments: a microscopy study. Microsc Res Tech. 2012; 75:1534-8. [DOI: 10.1002/jemt.22097] [PMID: 22791665]

9. Keller H. Corrosion determination techniques applied to endodontic instruments – irrigation solution systems. J Endod. 1982; 8:246-52. [DOI: 10.1016/S0099-2399(82)80334-8] [PMID: 6954542]

10. Casella G, Rosalbino F. Corrosion behaviour of NiTi endodontic instruments. Corros Eng Sci Techn. 2011; 46:521-3. [DOI: 10.1179/1478420911Y.159428167445]

11. Stokes WO, Di Fiore MP, Barsi TJ, Koerber A, Gilbert LJ, Lautenschläger PE. Corrosion in Stainless-Steel and Nickel-Titanium Files. J Endod. 1999; 25:17-20. [DOI: 10.1111/j.1365-2999-2009.00392-6] [PMID: 10196837]

12. Heling I, Roesken I, Dinur T, Szewc-Levine Y, Steinberg D. Bacterial and cytotoxic effects of sodium hypochlorite and sodium dichloroisocyanurate solutions in vitro. J Endod. 2001; 27:278-80. [DOI: 10.1016/S0099-2399-2001.00400-00009] [PMID: 11485267]

13. O’Hoy PYZ, Messer HH, Palamara JE. The effect of cleaning procedures on fracture properties and corrosion of NiTi files. Int Endod J. 2003, 36:2724-32. [DOI: 10.1046/j.1365-2919.2003.00709.x] [PMID: 14641435]

14. Jocpicic S, Živkovic S, Qian W, Zhang H, Haapasalo M. Tissue dissolution by sodium hypochlorite: Effect of concentration, temperature, agitation, and surfactant. J Endod. 2010; 36:1558-62. [DOI: 10.1016/j. iendo.2010.08.021] [PMID: 20728727]

15. Katayama H, Yamamoto M, Kodama T. Degradation behavior of protective rust layer in chloride solutions. Corrosion Engineering. 2000; 49:41-4. [DOI: 10.3323/jcorr1991.49.41]

16. Sarkar NK, Redmond W, Schwaninger B, Goldberg AJ. The chloride corrosion behaviour of four orthodontic wires. J Oral Rehabil. 1983; 10:121-8. [DOI: 10.1016/0099-2399(82)80334-8] [PMID: 6955425]

17. Bussinger PE. Corrosion in Stainless-Steel and Nickel-Titanium Files. J Endod. 1998; 24:1096-10. [DOI: 10.1016/S0099-2399-1998.00149.4] [PMID: 9823119]

18. Amiral CCF, Ormiga F, Gomes JACP. Electrochemical-induced dissolution of stainless steel files. Int Endod J. 2015; 48:137-44. [DOI: 10.1111/iej.12292] [PMID: 24702197]

19. Aboud LRL, Ormiga F, Gomes JACP. Electrochemical induced dissolution of fragments of nickel-titanium endodontic files and their removal from simulated root canals. Int Endod J. 2014; 47:155-62. [DOI: 10.1111/iej.12126] [PMID: 23659794]

20. Gasic J, Popovic J, Zivkovic S, Petrovic A, Barac R, Nikolic M. Ultrastructural analysis of the root canal walls after simultaneous irrigation of different sodium hypochlorite concentration and 0.2% chlorhexidine gluconate. Microsc Res Techniq. 2012; 75:1099-103. [DOI: 10.1002/jemt.22036] [PMID:22419366]

21. Matamala GR. Correlation model of the AISI 316 stainless steel pitting potential with cellulose bleach process variables. Corrosion. 1987, 43:97-100. [DOI: 10.5006/1.3583124]
22. Hülsmann M, Heckendorff M, Lennon A. Chelating agents in root canal treatment: mode of action and indications for their use. Int Endod J. 2003; 36:810-30. [DOI: 10.1111/j.1365-2591.2003.00754.x] [PMID: 14641420]

23. Fayyad DM, Mahran AH. Atomic force microscopic evaluation of nanostructure alterations of rotary NiTi instruments after immersion in irrigating solutions. Int Endod J. 2014; 47:567-73. [DOI: 10.1111/iej.12189] [PMID:24138190]

24. Reinhard G, Raddke M, Rammelt U. The role of the salts of weak acids in the chemical passivation of iron and steel in aqueous. Corrosion Sci. 1992; 33:307-13. [DOI: 10.1016/0010-938X(92)90154-U]

Received: 01/07/2015 • Accepted: 10/10/2015
Ultrasrunktarna analiza površine endodontskih instrumenata nakon potapanja u rastvore za irigaciju

Jelena Popović1, Goran Radenković2, Jovanka Gašić1, Aleksandar Mitić1, Marija Nikolić1, Radomir Barac1, Slavoljub Živković3
1Univerzitet u Nišu, Medicinski fakultet, Klinika za stomatologiju, Odeljenje za bolesti zuba i endodonciju, Niš, Srbija; 2Univerzitet u Nišu, Mašinski fakultet, Katedra za proizvodno-informacione tehnologije i menadžment, Niš, Srbija; 3Univerzitet u Beogradu, Stomatološki fakultet, Klinika za bolesti zuba i endodonciju, Beograd, Srbija.

Materijal i metode rada
U istraživanju je korisćeno 48 instrumenata od nikl-titanijuma i nerđajućeg čelika. Testiranje osetljivosti na koroziju je izvršeno potapanjem u rastvore NaOCl od 5,25%, CHX od 0,2% i EDTA od 17%. Analiza korozivnih oštećenja je urađena pomoću skening elektronskog mikroskopa (SEM) na različitim uveličanjima. Testiranje je izvršeno na terapevtičkim endodontskim instrumenatima od nikl-titanijuma i nerđajućeg čelika, ali razlika nije bila statistički značajna. Korozija nakon potapanja u 17% EDTA nije uočena ni kod jedne vrste endodontskih instrumenata. Primena 5,25% NaOCl i 0,2% CHX kao rastvora za irigaciju kanala korena može izazvati ozbiljne korozivne promene na površini instrumenata od nikl-titanijuma i nerđajućeg čelika.

Rezultati
Instrumeni od nikl-titanijuma su pokazali značajno veću osetljivost na koroziju nakon potapanja u 5,25% NaOCl u poređenju sa instrumentima od nerđajućeg čelika (p<0,001). Nakon potapanja u 0,2% CHX uočeno je korozivno oštećenje površine instrumenata od nikl-titanijuma i nerđajućeg čelika, ali razlika nije bila statistički značajna. Korozija nakon potapanja u 17% EDTA nije uočena ni kod jedne vrste endodontskih instrumenata.

Zaključak
Primena 5,25% NaOCl i 0,2% CHX kao rastvora za irigaciju kanala korena može izazvati ozbiljne korozivne promene na površini instrumenata od nikl-titanijuma i nerđajućeg čelika.

Ključne reči: korozija; endodontski instrumenti; nikl-titanijum; nerđajući čelik; SEM

UVOD
Frakture endodontskih instrumenata u kanalu korena tokom hemomehaničke preparacije je značajna komplikacija koja može ugroziti konačan ishod endodontskog lečenja. Jedan od važnih faktora koji izazivaju zamor materijala endodontskog instrumenata je površinska korozija. Ultrasrunktarna analiza površine endodontskih instrumenata od nikl-titanijuma i nerđajućeg čelika nakon potapanja u rastvore za irigaciju kanala korena zuba. Frakcija endodontskih instrumenata u kanalu korena tokom hemomehaničke preparacije je značajna komplikacija koja može ugroziti konačan ishod endodontskog lečenja. Jedan od važnih faktora koji izazivaju zamor materijala endodontskog instrumenata je površinska korozija. Ultrasrunktarna analiza površine endodontskih instrumenata od nikl-titanijuma i nerđajućeg čelika nakon potapanja u rastvore za irigaciju kanala korena zuba. Cilj ovog rada je bio da se ispita ultrastruktura površine endodontskih instrumenata od nerđajućeg čelika i nikl-titanijuma (Ni-Ti) nakon potapanja u najčešće upotrebljavane rastvore za irigaciju kanala korena zuba.

Materijal i metode rada
U istraživanju su korišćeni ručni endodontski instrumenti od Ni-Ti („I-FLEX“, „IMD“, SAD) i nerđajućeg čelika (NTI-Kahla GmbH, Nemačka). Novi instrumenti su u izradi iz bakra i, radi usavršavanja njihove strukturalne kapacitete, lako je instaliran i ugrađen u ultrazvučnu aplikaciju. Frakcija endodontskih instrumenata u kanalu korena tokom hemomehaničke preparacije je značajna komplikacija koja može ugroziti konačan ishod endodontskog lečenja. Jedan od važnih faktora koji izazivaju zamor materijala endodontskog instrumenata je površinska korozija. Ultrasrunktarna analiza površine endodontskih instrumenata od nikl-titanijuma i nerđajućeg čelika nakon potapanja u rastvore za irigaciju kanala korena zuba.

Ključne reči: korozija; endodontski instrumenti; nikl-titanijum; nerđajući čelik; SEM
na Katedri za proizvodno-informacione tehnologije Mašinskog fakulteta Univerziteta u Nišu i na Katedri za fizičku hemiju Tehnološko-metaluroškog fakulteta Univerziteta u Beogradu.

Nakon elektrohemijskih ispitivanja (objavljeni rezultati) [6], instrumenti su pripremani za skening-elektronskom mikroskopsko ispitivanje. Da bi se uzorci adekvatno podešavali i pričvrstili za cilindrične nosače, a radni delovi sa sečivima i navojima pričvršćeni su u aparat za jonsko raspršivanje (JEOL JSM-5300). Tako pripremljeni uzorci su posmatrani na skening-elektronmikroskopu (SEM) (JEOL JSM-5300). SEM ispitivanje je obavljeno na Institutu za biomedicinska istraživanja Medicinskog fakulteta u Nišu.

Ultragutovina površinske korozije je ocenjena modifikovanoj varijanci po Linsujvanontu (Linsuvanont) i saradnicima [7]: ocena 3 je označavala kontinuirano prelazeću površinu celog radnog dela instrumenta; ocena 2 je jasno ograničena korozivna polja; ocena 1 – pojedinačne korozivne jamice; ocena 0 – bez korozivnih promena. Površina radnih delova instrumenata je posmatrana na tri nivoa (apeksni, srednji i koronarni). Na svakom nivou instrument je ocenjen odgovarajućom ocenom, a konačni rezultat predstavlja srednju vrednost ocena sve tri trećine pojedinačno za svaki instrument i u okviru grupa. Statistička analiza je urađena uključujući test t-statički značajnosti s uslovom p<0,05.

REZULTATI

Ultrstrukturna analiza instrumenata je pokazala da su nacin izradbe određenih korozivnih promena nastale na Ni-Ti instrumentima nakon potapjanja u rastvor 5,25% NaOCl. Veoma erozivna površina metala je zapažena na Ni-Ti instrumenata i smanjuje efikasnost sečenja i otpornost na korozionalne promene [8]. Kompleksne hemijske i elektrohemijske procese koje se dešavaju tokom preparacije kanala korena, ali i kao sredstvo za natapanje instrumenata u procesu proizvodnje instrumenata, odnosno kvalitet kontrole kovanja, čime je potvrđeno da na pojavu korozije može uticati pravac postupka. Negativan uticaj sterilizacije na pojavu korozije su pokazali u ispitivanju određenih rastvora za irigaciju. Mnogi rastvori za sterilizaciju korozivni su za mnoge vrste endodontskih instrumenata i smanjuju efikasnost sečenja i otpornost na korozionalne promene [9].

Hemomehanička preparacija kanala korena je od suštinske važnosti za uspeh endodontskog lečenja i podrazumeva postupke čišćenja i oblikovanja endodontskih instrumenata i rastvora za sterilizaciju. Mnogi rastvori za sterilizaciju korozivni su za mnoge vrste endodontskih instrumenata i rastvora za sterilizaciju. Ukoliko instrumenti od nerđajućeg čelika pokazuju značajno veću osetljivost na potapanje u 5,25% NaOCl u odnosu na 0,2% CHX

DISKUSIJA

Hemomehanička preparacija kanala korena je od suštinske važnosti za uspeh endodontskog lečenja i podrazumeva postupke čišćenja i oblikovanja endodontskih instrumenata i rastvora za sterilizaciju. Mnogi rastvori za sterilizaciju korozivni su za mnoge vrste endodontskih instrumenata i rastvora za sterilizaciju. Ukoliko instrumenti od nerđajućeg čelika pokazuju značajno veću osećaj na potapanje u 5,25% NaOCl u odnosu na 0,2% CHX

Za korozivno ponašanje legura ni-kl-titanijuma i nerđajućeg čelika, kao materijala od kojih se najčešće izrađuju endodontski instrumenti, mogu uticati brojni faktori. Stouks (Stokes) i saradnici [11] su u ispitivanju korozivnog efekta novih, nekorišćenih Ni-Ti instrumenata pet različitih rastvorova na osećaj instrumenta i smanjenjem efikasnosti sečenja i otpornosti na korozivne promene [9].

Ni-Ti instrumenti su pripremani za skening-elektronmikroskopsko ispitivanje. Da bi se uzorci adekvatno podešavali i pričvrstili za cilindrične nosače, a radni delovi sa sečivima i navojima pričvršćeni su u aparat za jonsko raspršivanje (JEOL JSM-5300). Tako pripremljeni uzorci su posmatrani i analizirani na skening-elektronskom mikroskopu (SEM) (JEOL JSM-5300). SEM ispitivanje je obavljeno na Institutu za biomedicinska istraživanja Medicinskog fakulteta u Nišu. Statistička analiza je urađena uključujući test t-statički značajnosti s uslovom p<0,05.
od Ni-Ti i nerđajućeg čelika na potapanje u NaOCl je dokazano u brojnim studijama [1, 5, 13]. U istraživanju Stouksa i saradnika [11] korozija endodontskih instrumenata je vizuelno dokazana nakon potapanja u rastvor NaOCl u koncentraciji od 5,25%. Uočena je značajna razlika između različitih proizvođača, ali nije bilo statistički značajne razlike između instrumenata od Ni-Ti i nerđajućeg čelika. Beruti (Berutti) i saradnici [4] su u svojoj studiji dokazali da su instrumenti potapani u NaOCl imali značajno smanjenu otpornost na lomljenje usled pojave ranog cikličnog zamora. Pojava neočekivane frakture ovih instrumenata bila je značajno veća u odnosu na instrumente kontrolne grupe. SEM analizom u oblasti preloma instrumenata uočena su brojna ograničena korozivna polja, jamice i pukotine. Međutim, uticaj jona Cl- i F- na koroziju kod Ni-Ti i nerđajućeg čelika je iskorišćena u studijama Amarala (Amaral) i saradnika [18] i Abuda (Aboud) i saradnika [19] radi elektrohemijskog rastvaranja i uklanjanja frakturisanih endodontskih instrumenata iz kanala korena. U ovom istraživanju uočene su snažno erodirane kontinuirane korozivne površine duž radnih delova instrumenata posle potapanja u NaOCl u koncentraciji od 5,25%, što je u skladu s vrednostima piting potencijala dobijenim pri elektrohemijskom ispitivanju [6].

Hlorheksidin-glukonat (CHX) je sve češće upotrebljava- no sredstvo za irigaciju kanala korena zbog svojih antibakterijskih svojstava, koje može trajati i do 12 nedelja [20]. Kao irigans se koristi u opsegu koncentracija od 0,1% do 2%. Međutim, podaci iz literature ukazuju na mogućnost pojave površinske korozije instrumenata nakon potapanja u CHX [5, 6]. Korozivni potencijal CHX zavisi od njegovog pH vrednosti [7,8] jer se u koncentraciji od 4,5% potencijal potencijalna pH smanjuje na 7,5. U ovom istraživanju uočene su brojna ograničena korozivna polja, jamice i pukotine. Međutim, podaci iz literature ukazuju za mogućnost pojave površinske korozije instrumenata nakon potapanja u CHX [5, 6].

ZAKLJUČAK
Primena 5,25% rastvora NaOCl i 0,2% rastvora CHX za irigaciju kanala korena može izazvati ozbiljne korozivne promene na površini endodontskih instrumenata od Ni-Ti i nerđajućeg čelika. Primena rastvora EDTA u koncentraciji od 17% nije dovela do korozivnih promena kod ispitanih grupa instrumenata. Da bi se smanjio rizik od oštećenja instrumenata, preporučuje se da se instrumenti odmah potapanja u CHX uvede u koncentraciji od 0,2% i Nerđajućeg čelika posle potapanja u 0,2% CHX. Ovakva vidljiva oštećenja u vidu ograničenih polja i jamica su u skladu s vrednostima piting potencijala dobijenim pri elektrohemijskom ispitivanju [8].

Etilediamintetrasirćetna kiselina (EDTA) je helacijno sred- stvo koje se u endodonziji koristi u koncentracijama od 15% do 17%. Zbog svoje antibakterijskih svojstava može izazvati ozbiljne korozivne promene na površini endodontskih instrumenata od Ni-Ti i nerđajućeg čelika. Pri izazivanju rastvora EDTA u koncentraciji od 17% nije dovela do korozivnih promena kod ispitanih grupa instrumenata. Da bi se smanjio rizik od oštećenja instrumenata, preporučuje se da se instrumenti odmah po potapanju u CHX uvede u koncentraciji od 0,2% i Nerđajućeg čelika posle potapanja u 0,2% CHX. Ovakva vidljiva oštećenja u vidu ograničenih polja i jamica su u skladu s vrednostima piting potencijala dobijenim pri elektrohemijskom ispitivanju [8].

NAPOMENA
Deo rezultata je saopšten u vidu poster-prezentacije na „Dru- gom regionalnom okruglom stolu: vatrostanstvo, procesna in- dustrija i nanotehnologije Rosov PIN 2014”, koji je održan 23. i 24. oktobra 2014. godine na Fruškoj gori.