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THE EXAMINATION OF SENSITIVITY TO CORROSION OF NICKEL-TITANIUM AND STAINLESS STEEL ENDODONTIC INSTRUMENTS IN TOOTH ROOT CANAL IRRIGATING SOLUTIONS•

Article Highlights

- Corrosion of Ni-Ti and stainless steel endodontic files in irrigating solutions was examined
- Testing of sensitivity to corrosion was performed by dynamic potentiometric method
- Measurements were performed in 5.25% NaOCl, 0.2% CHX and 17% EDTA

Abstract

The application of irrigating solutions is essential in chemomechanical treatment of tooth root canal. However, chemical and electrochemical aggressiveness of the solutions, which directly act on the instruments, may damage their surface. The aim of this study was to investigate the sensitivity of the nickel-titanium (Ni-Ti) and stainless steel endodontic files to corrosive action of the sodium hypochlorite (NaOCl), chlorhexidine gluconate (CHX) and ethylenediamine tetraacetic acid (EDTA). Testing of sensitivity to corrosion of the instruments was performed by dynamic potentiometric method. Measurements were made in 5.25% NaOCl, 0.2% CHX and 17% EDTA. Ni-Ti instruments immersed in 5.25% NaOCl showed the most intensive corrosive changes and the lowest value of pitting potential of 1.1 V. Stainless steel instruments immersed in 5.25% NaOCl showed higher value of pitting potential of 1.5 V. Stainless steel instruments immersed in 0.2% CHX showed lower corrosive surface changes and higher value of pitting potential of 1.6 V, whereas Ni-Ti instruments immersed in 0.2% CHX showed the pitting potential of 1.9 V. The corrosion was not observed in both types of instruments after immersion in 17% EDTA. The use of 5.25% NaOCl and 0.2% CHX may cause severe surface corrosion of Ni-Ti and stainless steel endodontic files.

Keywords: corrosion, irrigating solutions, nickel-titanium, stainless steel, endodontic instruments.

Chemomechanical root canal preparation is essential during endodontic treatment and involves procedures of cleaning and shaping with endodontic instruments and irrigating solutions. The purpose of mechanical instrumentation is to obtain a continuous tapering funnel shape, flowing with the original canal from the coronal access to the apex. The functions of the irrigants are to act as media for removing debris, as lubricants, to dissolve smear layer from dentinal walls and to promote root canal sterility [1]. Many solutions, such as sodium hypochlorite (NaOCl), hydrogen peroxide (H_2O_2), citric acid ($C_6H_8O_7$), ethylenediamine tetraacetic acid (EDTA), chlorhexidine gluconate (CHX) and physiological saline, have been used for root canal irrigation [2]. Even though the benefits of irrigating solutions are essential for chemomechanical preparation, chemical and electrochemical aggressiveness of these solutions may damage the surface of the instruments [3].

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There are many literature data about susceptibility to corrosion of endodontic instruments in irrigating solutions [2,4]. The corrosion process could be activated during chemomechanical treatment, chemical disinfection of the instruments and sterilization [5]. Corrosion adversely affects the metallic surfaces by causing pitting and porosity, and decreases the cutting efficiency of endodontic files [6]. Several studies [7,8] have shown that corrosion of the endodontic files can degrade the mechanical properties and suddenly cause undesirable cracks that occur during root canal preparations.

The purpose of this study was to evaluate sensitivity to corrosion of nickel-titanium and stainless-steel endodontic files in most commonly used root canal irrigating solutions, NaOCl, CHX and EDTA.

EXPERIMENTAL

The study included 36 hand endodontic files divided according their type; 18 nickel-titanium (I--FLEX, IMD, USA) and 18 stainless-steel (NTI-Kahla GmbH, Germany). To remove all debris received from the manufacturers, the files were cleaned in an ultrasonic bath (JUS-S01, JEOL) with distilled water for 15 min at the frequency of 28 kHz immediately after taking them from the original packages. Each type of the instrument was divided into three groups according to the irrigant solutions examined in the study, so each group consisted of six files.

Measurements were performed in 5.25% NaOCl (prepared in the laboratory), 0.2% CHX (R4, Septodont, France, diluted to 0.2%) and 17% EDTA (prepared in the laboratory). All solutions used in this study were freshly prepared, and stored in adequate conditions. The corrosion behaviour was assessed using potentiodynamic method. 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 a way that only the working part of the instrument was immersed in the solution, whereas the base and the hand were above the solution. The instruments were immersed 15 s before the start of the potential rise and this time was set by the program. Anodic *E*–*I* polarization curves were recorded by using software Par Stat by means of the linear sweep technique (sweep rate 0.2 mV/s) in an air atmosphere at room temperature of 23±3 °C. 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 the metal and forming of the pits. The measurements were repeated six times for each solution and the each type of the file, and the results were given as mean values. Statistical analysis was carried out using Student's t-test and Mann-Whitney U test (SigmaStat statistical software). Electrochemical testings were performed at 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.

RESULTS AND DISCUSSION

The results of the study are shown in Table 1. The corrosion resistance was the lowest in the group of Ni-Ti instruments immersed in 5.25% NaOCl. The pitting potential was recorded at 1.1 V (Figure 1). Higher resistance to corrosion was observed in Ni-Ti instruments tested in 0.2% CHX. The measurements showed that the pitting potential was 1.9 V (Figure 2). Based on the obtained results it can be stated that Ni--Ti instruments immersed in 5.25% NaOCl and 0.2% CHX showed current increases and hence the tendency to pitting corrosion (Figures 1 and 2). Comparing the behavior of Ni-Ti instruments in 5.25% NaOCl and 0.2% CHX we can notice that NaOCl caused higher current increase that means less corrosion resistance. Statistical analysis showed that this difference was statistically significant ($P < 0.001$). On the contrary, Ni-Ti instruments immersed in 17% EDTA showed the highest resistance to corrosion. The rise of the current was not observed in the whole range of examined potentials and the value remained approximately constant (Figure 3).

Similar behavior was observed in the group of stainless steel instruments. The increase of current density was also high in 5.25% NaOCl (1.5 V) and

Table 1. Pitting potential values of the Ni-Ti and stainless steel instruments in tested irrigant solutions

Instrument	Irrigants	Mean±SD	Std. Error	C.I. of Mean	Max-Min	Median
Ni-Ti	5.25% NaOCl	1.1±0.089	0.037	±0.094	1.2-1.0	1.1
	0.2% CHX	1.9±0.141	0.058	±0.148	2.1-1.7	1.9
Stainless steel	5.25% NaOCl	1.5±0.141	0.058	±0.148	1.7-1.3	1.5
	0.2% CHX	1.6±0.063	0.026	±0.066	1.7-1.5	1.6

0.2% CHX (1.6 V), but the difference was not statistically significant (Figures 4 and 5). No significant increase of current in wide range of examined potentials was observed after immersion of stainless steel instruments in 17% EDTA (Figure 6).

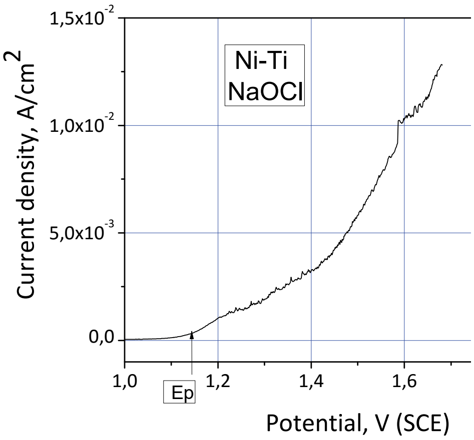

Figure 1. Potentiodynamic polarisation curve of the Ni-Ti file in 5.25% NaOCl.

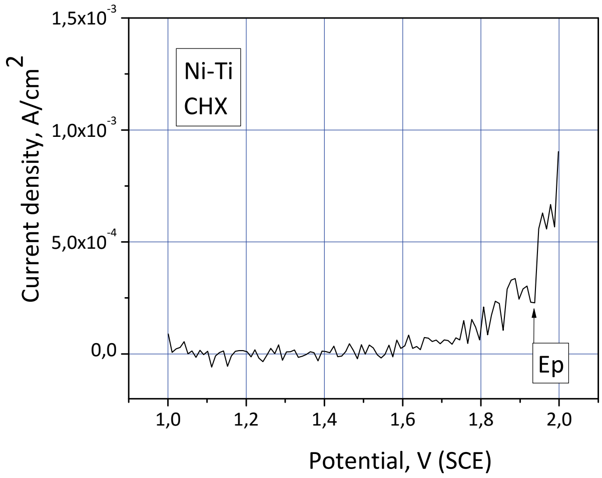

Figure 2. Potentiodynamic polarisation curve of the Ni-Ti file in 0.2% CHX.

According to the examined potentials in both types of the instruments, after immersion in 5.25% NaOCl Ni-Ti instruments showed less corrosion resistance compared to the stainless steel instruments and this difference was statistically significant ($P < 0.001$). After immersion in 0.2% CHX, Ni-Ti instruments showed higher resistance to corrosion compared to the stainless steel instruments, and the difference was statistically significant ($P < 0.01$).

The chemical mechanisms that occur either during instrumentation and irrigation of the root canal system, or after instrumentation (in procedures of instrument disinfection and sterilization), may cause corrosion and deterioration of the endodontic instruments [9-11]. Corrosion is a deterioration of a metal by chemical or an electrochemical reaction with its environment, and a technique that evaluates the electrochemical properties of the instrument-irrigating solutions system would seem most appropriate in studying corrosion [12]. Electrochemical techniques that are based on the electrode potential-current characteristics define the susceptibility of a metal to react with its environment [13].

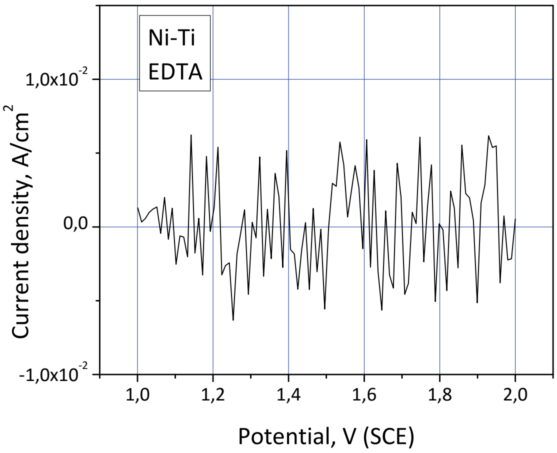

Figure 3. Potentiodynamic polarisation curve of the Ni-Ti file in 17% EDTA.

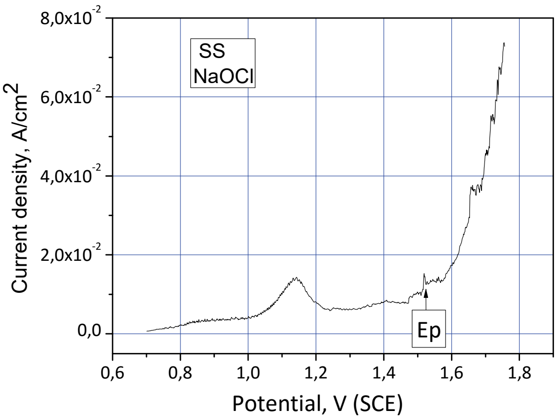

Figure 4. Potentiodynamic polarisation curve of the stainless steel file in 5.25% NaOCl.

During endodontic therapy, the most frequently used irrigant is sodium hypochlorite (NaOCl) in a concentration range of 0.5-6% [14]. It is an agent with wide spectrum of antimicrobial action and tissue dissolution capacity [15], which is also used as a pre-soaking solution in cleaning procedures of endodontic instruments after clinical use [9]. However, it is highly corrosive to metals and could cause corrosion of the endodontic files. Corrosion pattern involves pitting and potentially weakening of the structure of the instruments [16]. NaOCl contains active Cl^-, and it is well-known that Cl^- is an aggressive ion that generally increases corrosion rates [17]. This study showed that the corrosion rate of the endodontic files was high in 5.25% NaOCl. These results are in accordance with the results of earlier studies and confirm that the corrosion of endodontic files in NaOCl is possible. NaOCl is corrosive to many metals and selectively removes nickel from the Ni-Ti alloy [18]. Busslinger *et al.* [19] found measurable release of titanium when Lightspeed Ni-Ti files were immersed in NaOCl solution for 30 and 60 min. In the study of Stokes *et al.* [6] corrosion was visually observed on endodontic files after immersion in 5.25% NaOCl, there was significant difference in corrosion frequency between brands, but there was no difference between stainless steel and Ni-Ti instruments. Oztan *et al.* [2] revealed severe corrosion on the surface of the stainless steel endodontic instruments after immersion in 5.25% NaOCl, in accordance to O'Hoy *et al.* [9] who have shown evident signs of corrosion after overnight immersion of endodontic instruments in NaOCl. The fact that chloride and fluoride ions have negative effects on the corrosion resistance of stainless steel and Ni-Ti alloys is used in few investigations to promote electrochemical dissolution and removing endodontic instruments in cases where they are fractured in the root canal system [20,21].

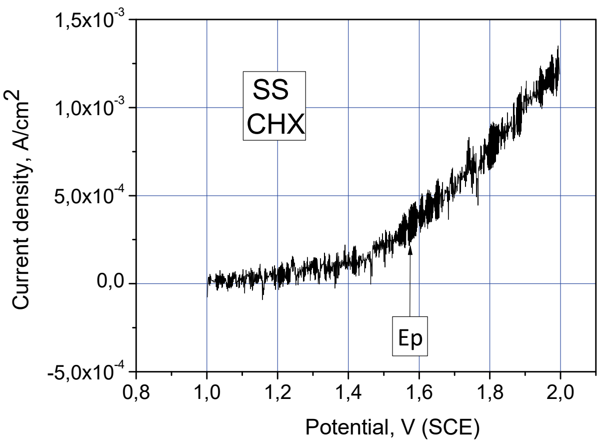

Figure 5. Potentiodynamic polarisation curve of the stainless steel file in 0.2% CHX.

Chlorhexidine gluconat (CHX) at concentrations 0.1-2% is a broad spectrum antimicrobial agent that is used during root canal irrigation. Its cationic structure provides a unique property, named substantivity. This prolonged antimicrobial activity in the root canal may last up to 12 weeks [22]. However, the literature data revealed that CHX can cause severe corrosion of endodontic instruments [2]. The results of this study confirmed that intensive surface corrosion can occur after immersion of the files in 0.2% CHX. According to the Matamala [23], this high rate of corrosive changes may depend on its acidic pH (5.72), as the acidic environment increases the corrosion rate.

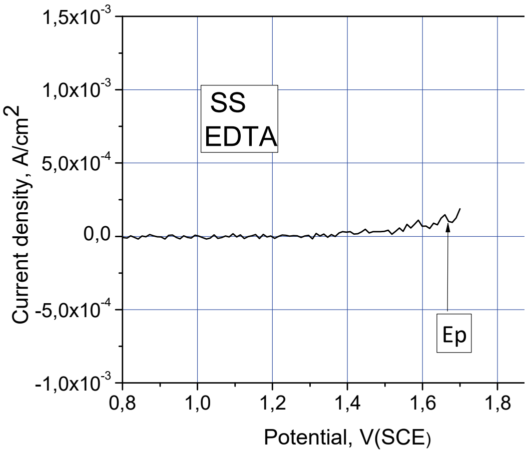

Figure 6. Potentiodynamic polarisation curve of the stainless steel file in 17% EDTA.

Ethylenediamine tetraacetic acid (EDTA) is the chelating irrigant with inorganic tissue dissolution capacity, and is used due to ability to lubricate and facilitate root canal instrumentation especially in preparation of narrow and curved root canals. In endodontics it is used as 15-17% solution [24]. The results of potentiodynamic test in this study did not reveal corrosion of endodontic files after immersion in 17% EDTA, and it was in accordance with literature data [2,4,25]. Öztan *et al.* [2] have reported the lowest corrosion rate of stainless steel endodontic files in 17% EDTA. They have stated that EDTA forms complexes with metal ions (Fe, Ni, Cr, Co, etc.) at pH values < 4. EDTA's ability to protect and passivate instruments is due to its ability to complex with iron to form an inhibiting barrier to oxidation and corrosion [26]. According to Darabara *et al.* [4], large molecules of R-EDTA have greater difficulty in concentrating and orienting the pit so as to increase the acidity to adequate values for trigger corrosion. Atomic force microscopic evaluation of Fayyad and Mahran [25] showed that immersion in 17% EDTA did not affect the surface roughness of the Ni-Ti endodontic instruments.

Endodontic files and reamers are generally accepted as reusable instruments. In purpose to eliminate the risk of infection transmission, these instruments need to be cleaned and sterilized thoroughly after clinical use [27]. However, these procedures could potentiate surface corrosion in irrigating solutions [28]. Casella and Rosalbino [29] confirmed that sterilization process had negative influence on the corrosion behavior of endodontic instruments, and the effect appears to be more dramatic for longer sterilization treatment periods. The presence of protein debris in form of ground tooth structure or collagen, with NaOCl solutions, could increase the severity of the surface attack on the instrument [13]. Stokes et al. [6] evaluated the corrosive effect of 5.25% NaOCl on stainless steel and Ni-TI files using five commercial brands. They reported that both the corroding and non-corroding files were present in the same packages. Those results showed that the severity of corrosive changes could also depend on manufacturing process and quality control.

CONCLUSION

The results of this study indicated that 5.25% NaOCl and 0.2% CHX, used as root canal irrigants, cause severe corrosion on the surface of the Ni-Ti and stainless steel endodontic files. The use of EDTA did not cause corrosion of the surface of both types of instruments. Due to the possibility of corrosion acting to deteriorate endodontic instruments, irrigants should be rinsed from files immediately after use and files should be replaced frequently.

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REFERENCES

[1] J. Walcott, V.T. Himel, J. Endod. **23** (1997) 217-224

[2] D.M. Öztan, A.A. Akman, L. Zaimoglu, S. Bilgiç, Int. Endod. J. **35** (2002) 655-659

[3] B.C. Sağlam, S. Koçak, M.M. Koçak, Ö. Topuz, Microsc. Res. Tech. **75** (2012) 1534-1538

[4] M. Darabara, L. Bourithis, S. Zinelis, G.D. Papadimitriou, Int. Endod. J. **37** (2004) 705-710

[5] K. Sood, B. Mohan, L. Lakshminarayanan, Endodontology **18** (2006) 34-41

[6] W.O. Stokes, M.P. Di Fiore, T.J. Barss, A. Koerber, L.J. Gilbert, P.E. Lautenschlager, J. Endod. **25** (1999) 17-20

[7] Y. Shen, M, Haapasalo, G.S. Cheung, B. Peng, J. Endod. **35** (2009) 129-132

[8] Y. Shen, G.S. Cheung, B. Peng, M. Haapasalo, J. Endod. **35** (2009) 133-136

[9] P.Y.Z. O'Hoy, H.H. Messer, J.E.A. Palamara, Int. Endod. J. **36** (2003) 724-732

[10] A.A. Yahya, K.W. Majida, AL-Hashimi, J. Bagh. College. Dentistry. **21** (2009) 53-59

[11] G. Spagnuolo, G. Ametrano, D. D'Antò, C. Rengo, M. Simeone, M. Riccitiello, M. Amato, Int. Endod. J. **45** (2012) 1148-1155

[12] G. Radenković, S.K. Zečević, Z. Cvijović, D.M. Dražić, J. Serb. Chem. Soc. **60** (1995) 51-59

[13] H.J. Mueller, J. Endod. **8** (1982) 246-252

[14] I. Heling, I. Rotstein, T. Dinur, Y. Szwec-Levine, D. Steinberg, J. Endod. **27** (2001) 278-280

[15] S. Stojicic, S. Zivkovic, W. Qian, H. Zhang, M. Haapasalo, J. Endod. **36** (2010) 1558-1562

[16] E. Berutti, E. Angelini, M. Rigolone, G. Migliaretti, D. Pasqualini, Int. Endod. J. **39** (2006) 693-699

[17] H. Katayama, M. Yamamoto, T. Kodama, Corros. Eng. **49** (2000) 41-44

[18] N.K. Sarkar, W. Redmond, B. Schwaninger, A.J. Goldberg, J. Oral. Rehabil. **10** (1983) 121-128

[19] A. Busslinger, B. Sener, F. Barbakow, Int. Endod. J. **31** (1998) 290-294

[20] L.R.L. Aboud, F. Ormiga, J.A.C.P. Gomes, Int. Endod. J. **47** (2014) 155-162

[21] C.C.F. Amaral, F. Ormiga, J.A.C.P. Gomes, Int Endod. J. **48** (2015) 137-144

[22] J. Gasic, J. Popovic, S. Zivkovic, A. Petrovic, R. Barac, M. Nikolic, Microsc. Res. Tech. **75** (2012) 1099-1103

[23] G.R. Matamala, Corrosion. **43** (1987) 97-100

[24] M. Hülsmann, M. Heckendorff, A. Lennon, Int. Endod. J. **36** (2003) 810-830

[25] D.M. Fayyad, A.H. Mahran, Int. Endod. J. **47** (2014) 567--573

[26] G. Reinhard, M. Radtke, U. Rammelt, Corros. Sci. **33** (1992) 307-313

[27] M.A. Saghiri, F. Garcia-Godoy, M. Lotfi, P. Mehrvazfar, M. Aminsobhani, S. Rezaie, K. Asgar, Scanning. **34** (2012) 309-315

[28] X.R. Nóvoa, B. Martin-Biedma, P. Varela-Patiño, A. Collazo, A. Macías-Luaces, G. Cantatore, M.C. Pérez, F. Magán-Muñoz, Int. Endod. J. **40** (2007) 36–44

[29] G. Casella, F. Rosalbino, Corros. Eng. Sci. Technol. **46** (2011) 521-523.

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NAUČNI RAD

ISPITIVANJE OSETLJIVOSTI ENDODONTSKIH INSTRUMENATA OD NIKL-TITANIJUMA I NERĐAJUĆEG ČELIKA NA KOROZIJU U RASTVORIMA ZA IRIGACIJU KANALA KORENA ZUBA

Primena sredstava za irigaciju kanala korena zuba je od suštinskog značaja u endodontskoj terapiji. Međutim, hemijska i elektrohemijska agresivnost ovih rastvora, koji direktno deluju na instrumente, može oštetiti njihovu površinu. Cilj istraživanja je bilo ispitivanje osetljivosti endodontskih turpija od nerđajućeg čelika i nikl-titanijuma (Ni-Ti) na koroziono delovanje natrijum-hipohlorita (NaOCl), hlorheksidin-glukonata (CHX) i etilendiamin tetrasirćetne kiseline (EDTA). Ispitivanje otpornosti instrumenata na koroziju je izvedeno potenciodinamičkom metodom. Merenje je izvedeno u rastvorima 5,25% NaOCl, 0,2% CHX i 17% EDTA. Najintenzivnije korozione promene i najnižu vrednost piting potencijala od 1.1 V su pokazali Ni-Ti instrumenti potapani u 5,25% NaOCl. Višu vrednost piting potencijala od 1.5 V su pokazali instrumenti od nerđajućeg čelika posle potapanja u 5,25% NaOCl. Manji intenzitet korozionih promena i piting potencijal od 1.6 V pokazali su instrumenti od nerđajućeg čelika potapani u 0,2% CHX, dok su Ni-Ti instrumenti potapani u 0,2% CHX pokazali vrednost piting potencijala od 1.9 V. Korozija nije zapažena kod obe vrste instrumenata nakon potapanja u 17% EDTA. Primena 5,25% NaOCl i 0,2% CHX može izazvati ozbiljnu koroziju površina endodontskih turpija od nerđajućeg čelika i nikl-titanijuma.

Ključne reči: korozija, irigacioni rastvori, nikl-titanijum, nerđajući čelik, endodontski instrumenti.