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Effects of sodium hypochlorite on corrosion of the rotary nickel-titanium endodontic instruments – SEM analysis

Утицај натријум хипохлорита на појаву корозије ротирајућих нити инструмената – СЕМ анализа

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SUMMARY

Introduction/Objective of this study is to use SEM analysis to examine surfaces of new and same sets of Ni-Ti instruments after canal preparations, to check their susceptibility to corrosion.

Methods In this study, we used five different endodontic Ni-Ti instruments: K3, Mtwo, ProTaper Universal, HyFlex and BioRaCe. Instruments were analyzed before and after preparation of canals of different curvature, using SEM (150-2000X).

Results Corrosion of the working part was observed in 5.5% of new Ni-Ti instruments of the K3 system (apical and middle segment), in 5.5% of Mtwo instruments (apical third) and in 11.1% of ProTaper Universal systems (apical and middle third). Corrosion was not observed on the new instruments of the HyFlex and BioRaCe kits. After instrumentation, disinfection and sterilization, corrosion was observed in all sets of K3 and ProTaper Universal systems and in all HyFlex instruments of the first group. Corrosion was observed in the HyFlex system in the second group in 16.7% of instruments (apical and middle third) and in the third 83.3% in the apical and 66.7% in the middle segment. In the Mtwo set, corrosion was observed in 16.7% of instruments in the first (apical and middle third), in the second group in 33.3% of instruments in the apical part and 50% in the middle third, while in the third group, corrosion was observed in 16.7% of instruments in the middle third of instruments.

Conclusion Rotary Ni-Ti instruments K3 and ProTaper Universal are susceptible to corrosion in a very high percentage. Ni-Ti systems with post-heat treatment of the working part (HyFlex) are somewhat more resistant to corrosion, while in Ni-Ti systems with electropolished surface (BioRaCe), corrosion is not observed.

Keywords: Corrosion; Ni-Ti file; Scanning electron microscopy (SEM)

САЖЕТАК

Увод/Циљ Циљ истраживања је био да се применом СЕМ-а анализирају површине некоришћених и истих сетова никл-титанијмских (НиТи) ротирајућих инструмената након препарације канала, односно да се провери њихова подложност за појаву корозије.

Методе У истраживање је укључено пет различитих сетова машинских ендодонтских НиТи инструмената: К3, Мтwо, Про Тапер Универсал, Ху Флех и БиоРаЦе. Инструменти су анализирани пре употребе и након препарације канала различите повијености, помоћу СЕМ-а (150-2000×). Сваки сет инструмената је коришћен за обраду 10 канала у три експерименталне групе (прави, благо и изразито повијени канали).

Резултати Корозија радног дела уочена је код 5,5% нових НиТи инструмената К3 система (апикални и средњи сегмент), у 5,5% МТwo инструмента (апикална трећина) и у 11,1% ПроТапер Универсал система (апикална и средња трећина). Корозија није уочена на новим инструментима ХуФлех и БиоРаЦе сетова. Након инструментације, дезинфекције и стерилизације корозија је примећена код свих сетова К3 и ПроТапер Универсал система и код свих ХуФлех инструмената прве групе. Корозија је уочена код ХуФлех система у другој групи 16,7% инструмената (апикална и средња трећина) а у трећој 83,3% у апикалном и 66,7% у средњем сегменту. Код Мтwо сета, корозија је уочена у 16,7% инструмената прве (апикална и средња трећина), у другој групи код 33,3% инструмената у апикалном делу и 50% у средњој трећини, док је у трећој групи, корозија примећена у 16 7% инструмената у средњој трећини инструмената.

Закључак Ротирајући НиТи инструменти К3 и ПроТапер Универсал су подложни корозији у веома високом проценту. НиТи системи са накнадном термичком обрадом радног дела (ХуФлех) су нешто отпорнији на корозију, док код НиТи система са електрополираном површином (БиоРаЦе), коризија није уочена.

Кључне речи: Корозија, Ротирајући НиТи инструменти, Скенинг Електронска Микроскопија (СЕМ)

INTRODUCTION

During the last decades, machine instrumentation of root canals with the use of rotary Ni-Ti instruments has become a standard clinical procedure and this Ni-Ti instruments, have enabled easier and faster instrumentation with a predictable outcome [1, 2]. The fact is that various factors during mechanical instrumentation of the canal can affect the occurrence of deformations and unexpected fractures of Ni-Ti instruments (anatomomorphological characteristics of the canal, inadequate choice of endodontic instruments and preparation techniques, means and techniques of irrigation, knowledge, expertise and experience), which significantly frustrates many practitioners and limits the use of these instruments in certain clinical indications [3]. One of the factors that can affect the efficiency and safety of their application is the occurrence of corrosion on the working part of NiTi rotary instruments [4].

Analyzing the surface of new, unused Ni-Ti instruments, many studies have shown that on the surface of their working part there are numerous defects (pitting, fretting, metal strips) [5, 6, 7]. The reason for this is the significantly more complex process of making rotating Ni-Ti instruments than the making of steel instruments, which often leads to irregularities on the surface of new Ni-Ti instruments [6, 7]. These changes represent centers with dislocation of the crystal structure that can compromise the cutting efficiency of the instruments and become sites for potential corrosion. Also, these points represent the sites of initiation of defects and may contribute to the degradation of mechanical properties and the appearance of micro or complete fractures during the clinical use of Ni-Ti instruments [8]. The presence of numerous defects on the working part of the instruments as a consequence of the production process often leads to corrosive effects on the used instruments.

Corrosion, as an oxidative reaction, leads to the release of electrons from metals, their movement towards the surface and the formation of molecular hydrogen [4]. It has been confirmed that the occurrence of corrosion can affect the reduced blade efficiency of rotary Ni-

Ti instruments, as well as the possible propagation of other defects [2].

Although it has been proven that there are several types of corrosion (uniform, galvanic, cracked, pitted, intergranular, selective corrosion corrosion, corrosion in the form of erosion and grooves, corrosion caused by material stress), pitting corrosion is the type of corrosion most common on Ni-Ti alloy surface [4]. The big problem with this form of corrosion is that the defects are difficult to detect, until the moment of exposure to aggressive ions (mainly chlorides) which further propagate this defect. In the mechanism of this type of corrosion the main role is played by the microstructure of the material and environmental conditions, such as Ph chloride concentration and temperature [4].

The appearance of corrosion on Ni-Ti instruments can be caused by the use of different irrigants during instrumentation, specific conditions of the oral environment (body temperature, saliva with salts and electrolytes, blood) as well as multiple cycles of chemical disinfection and sterilization [9]. The most commonly used irrigant, sodium hypochlorite (NaOCl), is highly corrosive to Ni-Ti alloy, ranging in concentrations from 1.2% to 5.25% [2, 10]. Namely, NaOCl selectively removes nickel from the surface of the instrument, and thus leads to the formation of microcracks, which negatively affect the physical and mechanical properties of rotary Ni-Ti instruments [2].

Although many studies have been conducted in the last two decades regarding the effect of NaOCl on Ni-Ti instruments, using microscopy and electrochemical analysis, quantitative data on corrosion of Ni-Ti rotary instruments are still scarce [2].

The aim of this research was to analyze the surfaces of new (unused) and the same sets of rotary Ni-Ti instruments after the preparation of the canals, ie to check their susceptibility to corrosion.

METHODS

The study included five sets of new rotary Ni-Ti instruments of different design: K3 (SybronEndo Co, USA), Mtwo (VDW, Munich, Germany), ProTaperUniversal (Dentsply Maillefer, Switzerland), HyFlex (Coltene Whaledent group, Switzerland) and BioRaCe (FKG DENTAIRE Swiss Dental Products, Switzerland) (Table 1).

The research was performed *in vitro* conditions, on permanent, multi-rooted teeth, extracted for various reasons after obtaining the consent of the Ethics Committee of the School of Dentistry University in Belgrade number 36/6 from 21.01. 2013. After extraction, the teeth were stored for two hours in 4% sodium hypochlorite solution, and until the beginning of the preparation, they were stored in physiological solution with 0.2% thymol. With the help of a high-speed elbow with water spray and a tungsten-carbide cylindrical drill with a rounded tip E 0153/012 (Dentsply / Maillefer, Ballaigues, Switzerland), the existing fillings and cariously changed tissues were removed, and a round drill with an extended handle E0123 / 014 (Dentsply) has done trepanation of the coronary chamber. With the diamond disk, the crown of the tooth was shortened to the level of 2 mm coronally from the enamel-cement border.

The final treatment of the walls of the access cavity and the coronal chamber was completed with a carbide conical drill with a passive tip Endo Z (Dentsply / Maillefer). Probing and assessment of the initial patency of the channel was determined by K-files of ISO 15 size (MicroMega, France) and the working length was determined to be 1 mm shorter than the length obtained by the appearance of the instrument at the top. An X-ray was performed on each canal and then the degree of bending for each canal was determined using an online protractor (*https://www.ginifab.com/feeds/angle_measurement/).*

The degree of canal curvature was determined by Schneider's radiographic technique [10] and based on that, the teeth were divided into three categories:

a) 50 straight canals - less than 10 °

b) 50 slightly bent canals - from 10 ° to 25 ° and

c) 50 strongly bent canals - over 25 °.

To mimic *in vivo* conditions, the apex was sealed with pink wax to simulate apical counter-pressure and prevent irrigation from leaking during instrumentation.

In order to achieve uniform experimental conditions, each instrument was used in ten canals or until the moment of its fracture (one set from all five examined threads of the system was applied for processing of 10 canals in each experimental group). The instrumentation of the canal was realized in accordance with the manufacturer's instructions, crown-down technique and application of X-Smart Endodontic Rotary Motor (Dentsply, Sirona, Maillefer, Ballaigues Salzburg, Austria).

As irrigants, after each instrument, in the amount of 5 cm^3, 2% NaOCl solution (CHLORAXID 2%, Cerkamed, Polska) and then Distilled water (Iva, Serbia) were used. Irrigants were applied using a plastic syringe and an endodontic irrigation needle with a closed tip and side openings (Side-vented needle, SmearClear, SybronEndo). Ethylenediamine tetra-acetic acid gel (Glyde-Dentsply, Maillefer, Switzerland) was used as a lubricant during the preparation, applied to the working part of the instruments. During the processing of the canal, each used instrument was carefully inspected with a magnifying glass, in order to detect any change (possible cracks, fractures, unscrewing of twist or other deformations).

The experimental protocol included:

1. SEM analysis, new unused instruments directly from the factory packaging, the apical and middle thirds of the instrument from two different directions were analyzed and three SE images were made for each surface of the instrument

2. preoperative preparation, cleaning in an ultrasonic bath using a mild disinfectant Orocid Multisept plus ("OCC", Switzerland) for 15 minutes

3. Instrumentation according to the proposed protocol with abundant irrigation (2% NaOCl

solution, Distilled water and EDTA)

4. Cleaning in an ultrasonic bath after use with a mild disinfectant (Orocid Multisept plus) for 15 minutes

5. Sterilization of used instruments performed in an autoclave (MELAG - VACUKLAV 23B +, Berlin, Germany) at 134ºC for five minutes.

6. SEM analysis of used instruments

For defect analysis, the following was inspected:

- 540 recordings of new instruments and 700 recordings of instruments after canal preparation
- Reconciliation of the results of the two researchers was performed by Cohen Kappa analysis

Statistical analysis

Statistical analysis the obtained data was performed using the Fisher test.

RESULTS

The results of the SEM analysis are shown in Tables 2 and 3 and Figures 1, 2, 3, 4, 5, and 6.

Corrosion of the working part of new Ni-Ti instruments was not observed in HyFlex and BioRaCe instruments, while in other Ni-Ti systems it was observed in a small percentage (Table 2).

In new Ni-Ti instruments, corrosion was observed in 5.5% of K3 system instruments (apical and middle segment), in 5.5% of Mtwo instruments (apical third) (Figure 1) and in 11.1% of ProTaper Universal system instruments (apical and middle third). Corrosion was not observed on the new sets of HyFlex and BioRaCe instruments (Table 2).

SEM analysis of Ni-Ti instruments after their use, cleaning and sterilization indicated the occurrence of corrosion in 62.8% of the analyzed instruments. After chemo-mechanical instrumentation, disinfection and sterilization, the presence of corrosion was not registered on the Ni-Ti instruments of the BioRaCe system. The presence of corrosion was observed on all analyzed Ni-Ti instruments (100%) K3 and ProTaper Universal system (apical and middle third), in all three experimental groups (Table 3).

In the first group, corrosion was observed on all instruments K3, ProTaper Universal and HyFlex systems (apical and middle third), and in Mtwo systems on 16.7% of instruments (apical and middle third). Corrosion was not observed in BioRaCe instruments. A statistically significant difference was observed between the K3, ProTaper Universal and HyFlex systems compared to the Mtwo and BioRaCe systems of the first experimental group ($p < 0.05$) (Table 3, Figure 2).

In the second experimental group, corrosion was observed on all analyzed Ni-Ti instruments K3 (Figure 3) and ProTaper Universal system (apical and middle third), in HyFlex set on 16.7% of instruments (apical and middle third), and in Mtwo set instruments, to 33.3% of instruments in the apical segment and 50.0% in the middle third. No corrosion was observed on BioRaCe instruments. A statistically significant difference was observed between K3 and ProTaper Universal systems compared to Mtwo, HyFlex and BioRaCe system ($p < 0.05$) (Table 3, Figure 4).

In the third experimental group, corrosion was observed on all analyzed NiTi instruments K3 and ProTaper Universal system (apical and middle third), in 83.3% of HyFlex set instruments in the apical and 66.7% in the middle segment (Figure 5). In the Mtwo set, corrosion was observed only in the middle third in 16. 7% of the instruments. A statistically significant difference in the occurrence of corrosion was observed between K3, ProTaper Universal and HyFlex systems in relation to Mtwo and BioRaCe system ($p < 0.05$) (Table 3, Figure 6).

Comparing the results of SEM analysis, it was found that corrosion is the only defect that shows a statistically significant difference between new and used instruments. This difference was found between new and used K3 and ProTaper Universal instruments in all groups, and between new and used HyFlex instruments in the first and third groups ($p < 0.05$).

DISCUSSION

The results of this study indicate a low prevalence of corrosion on K3, ProTaper Universal and Mtwo sets, as well as the absence of corrosion on HyFlex and BioRaCe system instruments. This result is in accordance with the statements indicating the biocompatibility and good corrosion resistance of Ni-Ti alloy [2, 11, 12].

Corrosion resistance of Ni-Ti alloy is based on the presence of a passive oxide film of titanium oxide on the surface which prevents the development of uniform corrosion. However, beneath this thin layer of titanium oxide (which also contains small amounts of nickel) is a nickel-rich sublayer that is responsible for corrosion [11]. It is believed that this surface oxide film of titanium oxide increases the stability of the surface layers of the alloy, protects against corrosion and ensures the stability of the material itself [13]. According to the results of research by Stokes and associates who examined the corrosive effect of new, unused Ni-Ti instruments from five different manufacturers, it was confirmed that the occurrence of corrosion on new instruments is influenced by the instrument production process, ie the quality of control of the manufacturing process [14].

The finding of a significantly higher percentage of corrosion of Ni-Ti rotating instruments after instrumentation, disinfection and sterilization is in line with the results of studies confirming that corrosion processes and mechanisms can be activated during chemo-mechanical treatment of channels and application of various organolytic and mineralolytic substances CHX, citric acid,…) which with their chemical and electrochemical potential can act on the surface structure of instruments [15]. It was found that changes in the surface of the working part of Ni-Ti instruments can occur as a result of chemo-mechanical treatment of instruments after instrumentation (cleaning, chemical disinfection or sterilization) [2, 9, 11].

The current results of studies on the impact of sterilization on the occurrence of corrosion are contradictory and there is no clear position on this issue. Multiple sterilization cycles can cause corrosive changes on the surface of Ni-Ti files due to changes in the surface layer of titanium oxide [16, 17]. However, it has also been observed that after sterilization, resistance to cyclic fatigue and torsional stress increases in certain types of Ni-Ti instruments because the sterilization process acts as a form of heat treatment [18]. Manufacturers recommend the mandatory use of gel lubricants during machine instrumentation, either directly on the active part of the Ni-Ti instrument or by application to the pulp chamber [9]. The most commonly used chelating, mineralolytic agent is ethylenediaminetetraacetic acid (EDTA, 15% to 17%), which according to the literature has no effect on the surface structure of Ni-Ti instruments [19, 20]. In the study of Fajad and Mahran, after immersing the instruments in a 17% solution of EDTA, there was no change in their surface structure [19]. According to research by Reinhard et al., EDTA protects and passivates the surface of NiTi instruments by forming complexes with metal ions (at pH values less than 4), thus creating an inhibitory barrier to oxidation and corrosion [20].

Experimental evidence does not support the use of these gels, as they not only do not reduce the friction between the instrument and the dentinal canal, but in some cases even increase it [13]. Aqueous solutions (or even distilled water) are much more useful, as they wash the dentinal detritus from the grooves of the instruments more efficiently [9, 13]. The use of NaOCl solution during instrumentation is standard because in addition to acting on bacteria and dissolving tissue debris, it is also very effective as a lubricant. NaOCl was confirmed to be highly corrosive to Ni-Ti alloy (in the concentration range 1.2% to 5.25%). A lower NaOCl concentration (1%) does not lead to corrosion and does not affect torsional and cyclic resistance after a cumulative exposure of 2.5 hours, while a longer exposure of 18 hours indicates clear signs of corrosion [21, 22].

The problem that arises during the disinfection process and the long-term complete immersion of the instruments in the sodium hypochlorite solution, arises due to the metallurgical characteristics of the instruments. The handle of the instrument is usually made of different metal in relation to the working part, so the presence of two metals in the solution can affect the release of ions and the creation of galvanic reactions that can accelerate corrosion [21]. It was confirmed that Ni-Ti alloy corrodes in NaOCl solution with high pH values (pH 12.3) due to galvanization (due to gilding on the handle). It is believed that the corrosion resistance of Ni-Ti alloy can be increased by lowering the pH value of the solution to about 10, because then passive oxides, TiO2 and NiO2 are formed [23].

The occurrence of corrosion on Ni-Ti instruments in this study could be explained by the fact that the application of various chemical procedures before or during instrumentation (disinfection, sterilization and irrigation) can cause corrosion or deepening of existing corrosive defects [22, 23]. Although the surface of rotating Ni-Ti instruments is usually covered with a protective film of titanium oxide, this layer can be easily disturbed and damaged during instrumentation and contact of the instrument with the root canal wall [23, 24, 25].

O'Hoy showed strong corrosion after immersion of instruments in NaOCl solution and Yokoyama pointed to corrosion as the main reason for the occurrence of Ni-Ti instrument fracture due to stress due to cyclic fatigue [22, 26]. Berutti pointed to a significant effect of 5% NaOCl solution (within five minutes) on the appearance of pits and cracks on the surface of NiTi instruments and Peters proved a decrease in resistance to cyclic fatigue of Race and ProFile instruments after their exposure to NaOCl [27, 28]. The reason for such contradictory results is probably a consequence of different methodological procedures (different immersion times of instruments, different concentration of solution, different exposure during irrigation, cleaning and disinfection) [21–28].

The results of the study by Darabara et al indicated that continuous irrigation with 2.5% NaOCl solution does not lead to corrosion of stainless-steel instruments and Ni-Ti instruments, but that mechanical rather than corrosive factors are responsible for the fracture [29].

The results of studies by Shahi et al indicated the resistance of Mtwo instruments to NaOCl solution. Immersion of Ni-Ti instruments in 2.5% NaOCl solution for 12 to 48 hours did not indicate significant changes in the working part of the instruments, which is in accordance with the findings of this study [30].

The BioRaCe system of Ni-Ti rotating instruments is subjected to an electropolishing procedure during final processing, which reduces the possibility of surface irregularities and increases resistance to corrosion and cyclic fatigue, which is also in accordance with the results of this study [2, 9]. Shahi hypothesized that corrosion occurs due to manufacturing defects on the surface of instruments that amplify the effect of cyclic fatigue and change the fracture mechanism due to cyclic fatigue into a corrosive fracture [30].

Improving corrosion resistance, manufacturers are trying to achieve by additional surface treatment of the working part by electropolishing, heat treatment, implementation, physical deposition or coating of various elements [2, 9].

CONCLUSION

The results of this study showed that the rotary Ni-Ti instruments of the K3 and Pro aper Universal systems are subject to corrosion in a very high percentage. Ni-Ti heat-treated systems are somewhat more resistant (HyFlex), while corrosion is not observed in instruments with electropolished surface (BioRaCe).

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REFERENCES

1. Živković S, Nešković J, Popović-Bajić M, Medojević-Jovanović M. The efficiency of canal cleaning with reciprocating movements instruments-SEM study. Srp Arh Celok Lek. 2020;148(3-4):148-52. https://doi.org/10.2298/SARH190412002Z
2. Lin JHH, Karabucak B, Lee SM. Effect of sodium hypochlorite on conventional and heat-treated nickel-titanium endodontic rotary instruments. An in vitro study. J Dent Sci. 2020 In press. https://doi.org/10.1016/j.jds.2020.08.015
3. de Carvalho KKT, Petean IBF, Silva-Sousa AC, de Camargo RV, Mazzi-Chaves JF, Silva-Sousa YTC, Sousa-Neto MD. Impact of several NiTi-thermally treated instrumentation systems on biomechanical preparation of curved root canals in extracted mandibular molars. Int Endod J. 2022 Jan;55(1):124-136. https://doi.org/10.1111/iej.13649. Epub 2021 Nov 4. PMID: 34687053.
4. Koch G, Varney J, Thompson N, Moghissi O, Gould M, Payer J. International measures of prevention, application, and economics of corrosion technologies study: Technical report. Houston, TX: NACE International. 2016. Available from: http://impact.nace.org/documents/Nace-International-Report.pdf
5. Eggert C, Peters O, Barbakow F. Wear of Nickel-Titanium Lightspeed Instruments Evaluated by Scanning Electron Microscopy. J Endod. 1999;25(7):494-7. https://doi.org/10.1016/S0099-2399(99)80289-1; PMID: 10687515
6. Martins JNR, Silva EJNL, Marques D, Pereira MR, Vieira VTL, Arantes-Oliveira S, Martins RF, Braz Fernandes F, Versiani M. Design, Metallurgical Features, and Mechanical Behaviour of NiTi Endodontic Instruments from Five Different Heat-Treated Rotary Systems. Materials. 2022;15(3):1009. https://doi.org/10.3390/ma15031009
7. Gavini G, Santos MD, Caldeira CL, Machado MEL, Freire LG, Iglecias EF, Peters OA, Candeiro GTM. Nickel-titanium instruments in endodontics: a concise review of the state of the art. Braz Oral Res. 2018;32(suppl.1):e67. https://doi.org/10.1590/1807-3107bor-2018.vol32.0067; PMID: 30365608
8. Thompson SA. An overview of nickel-titanium alloys used in dentistry. Int Endod J. 2000;33(4):297-310. https://doi.org/10.1046/j.1365-2591.2000.00339.x PMID: 11307203
9. Mohammadi Z, Kinoshita J, Manabe A, Kobayashi M, Shalavi S, Jafarzadeh H. Corrosion of Ni-Ti Rotary Instruments: A Review. J Dent Mater Tech. 2019;8(4):215-20. https://doi: 10.22038/jdmt.2019.14144;
10. Schneider SW. A comparison of canal preparations in straight and curved root canals. Oral Surg Oral Med Oral Pathol. 1971;32(2):271-5. https://doi.org/10.1016/0030-4220(71)90230-1; PMID: 5284110
11. Zanza A, D'Angelo M, Reda R, Gambarini G, Testarelli L, Di Nardo D. An Update on Nickel-Titanium Rotary Instruments in Endodontics: Mechanical Characteristics, Testing and Future Perspective—An Overview. Bioengineering. 2021; 8(12):218. https://doi.org/10.3390/bioengineering8120218
12. Torres Sánchez DL. Magnetic properties of NiTi/(Ni, Co) heterostructures [PhD dissertation]. São Paulo: Physics Institute, University of São Paulo; 2018. https://doi: 10.11606/T.43.2018.tde-28082018-230553;
13. Dobrzański LA, Dobrzański LB, Dobrzańska-Danikiewicz AD, Dobrzańska J. Nitinol Type Alloys General Characteristics and Applications in Endodontics. Processes. 2022; 10(1):101. https://doi.org/10.3390/pr10010101 ;
14. Stokes WO, Di Fiore MP, Barss TJ, Koerber A, Gilbert LJ, Lauten- schlager PE. Corrosion in Stainless-Steel and Nickel-Titanium Files. J Endod. 1999;25(1):17-20. https://doi.org/10.1016/S0099-2399(99)80392-6; PMID: 10196837;
15. Sarwar H, Naeem MM, Shabbir S, Riaz S, Afaq H, Shabbir J. Effect of hydrogenperoxide on cyclic fatigue resistance of NiTi endodontic files. J Pak Dent Assoc 2020;29(4):246-248. DOI: https://doi.org/10.25301/JPDA.294.246;
16. Palekar A, Vajpayee A, Biradar B. Recent advances in metallurgy and design of rotary endodontic instruments: a review. Int J Dent Mater 2020;2(2): 52-59. DOI: http://dx.doi.org/10.37983/IJDM.2020.2204 ;
17. Cassol LG, Kowalczuck A, Carneiro E, Westphalen VPD, Laurindo CAH, da Silva Neto UX. Evaluation of fluoride and sodium hypochlorite solutions during the electrochemical dissolution of conventional NiTi instruments and Gold thermomechanically treated NiTi instruments. Int Endod J. 2020;53(4):513-8. https://doi.org/10.1111/iej.13244; PMID:31691303
18. Costa TD, Silva EDFE, Caetano PL, et al. Corrosion resistance assessment of nickel-titanium endodontic files with and without heat treatment. Restor Dent Endod. 2020;46(1):e6. Published 2020 Dec 28. https://doi:10.5395/rde.2021.46.e6;
19. 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(6):567-73. https://doi.org/10.1111/iej.12189; PMID:24138190;
20. Reinhard G, Radtke M, Rammelt U. On the role of the salts of weak acids in the chemical passivation of iron and steel in aqueous solutions. Corros Sci. 1992;33(2):307-13. https://doi.org/10.1016/0010-938X(92)90154-U;

21. Bonaccorso A, Tripi TR, Rondelli G, Condorelli GG, Cantatore G, Schäfer E. Pitting Corrosion Resistance of Nickel-Titanium Rotary Instruments with Different Surface Treatments in Seventeen Percent Ethylenediaminetetraacetic Acid and Sodium Chloride Solutions. J Endod. 2008;34(2):208-11. https://doi.org/10.1016/j.joen.2007.11.012; PMID:18215684;
22. O'Hoy PYZ, Messer HH, Palamara JEA. The effect of cleaning procedures on fracture properties and corrosion of NiTi files. Int Endod J. 2003;36(11):724-32. https://doi.org/10.1046/j.1365-2591.2003.00709.x; PMID:14641435;
23. Hryniewicz T, Rokicki R. Modification of Nitinol Biomaterial for Medical Applications. World Scientific News. 2018:35-58.
24. Yon MJ, Tang MH, Cheung GS. Defects and Safety of NiTi Root Canal Instruments: A Systematic Review and Meta-Analysis. Front. Dent. Med. 2021;2:747071. https://doi: 10.3389/fdmed.2021.747071;
25. Busslinger A, Sener B, Barbakow F. Effects of sodium hypochlorite on nickel-titanium Lightspeed instruments. Int Endod J. 1998;31(4):290-4. https://doi.org/10.1046/j.1365-2591.1998.00149.x ; PMID:9823119;
26. Yokoyama K, Kancko K, Yabuta E, Asaoka K, Sakai J. Fracture of nickel-titanium superelastic alloy in sodium hypochlorite solution. Mater Sci Eng A. 2004;369(1-2):43-8. https://doi.org/10.1016/j.msea.2003.10.320;
27. 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(9):693-9. https://doi.org/10.1111/j.1365-2591.2006.01134.x; PMID:16916358;
28. Peters OA, Roehlike JO, Baumann MA. Effect of immersion in sodium hypochlorite on torque and fatigue resistance of nickel-titanium instruments. J Endod. 2007;33(5):589-93. https://doi.org/10.1016/j.joen.2007.01.007; PMID:17437879;
29. 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 (10):705-10. https://doi.org/10.1111/j.1365-2591.2004.00866.x; PMID:15347296;
30. Shahi S, Mokhtari H, Rahimi S, Shiezadeh V, Ashasi H, Abdolrahimi M, Foroughreyhani M. Electrochemical corrosion assessment of RaCe and Mtwo rotary nickle-titanium instruments after clinical use and sterilization. Med Oral Patol Oral Cir Bucal. 2012;17(2):e331-6. https://doi.org/10.4317/medoral.17413; PMID:22143690 PMCID:PMC3464121

Table 1. Basic characteristics of tested sets of rotary Ni-Ti instruments

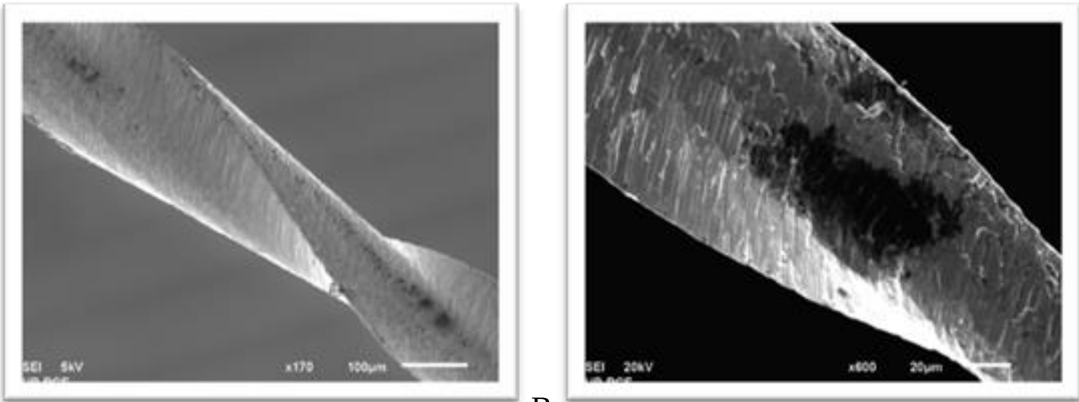
Instrument manufacturer	**Design specifics**	**Diameter**	**Taper**	**The process production**
K3 SybronEndo	triple blades with, positive angle and asymmetric radial surfaces	25	0.12–0.02	micromilling conventional Ni-Ti
Mtwo VDW	S-shape with two active cutting surface angle	10–35	0.04 0.05 0.06	micromilling conventional Ni-Ti
ProTaperUniversal Dentsplay-Sirona	convex triangle variable progressive taper along the instrument	17–30	regressive taper	micromilling conventional Ni-Ti
HyFlex CM Coltene	double Hedstrom design with positive rake angle	20–40	0.04 0.06 0.08	micromilling CM-wire
BioRaCe FKG	triangular with alterations of the cutting edges along the instrument	15–40	0.04 0.05 0.06 0.08	micromilling conventional Ni-Ti Electropolished surface

Table 2. Presence of the signs of corrosion on new rotary Ni-Ti instruments

K3		**Mtwo**		**ProTaper Universal**		**HyFlex**		**BioRaCe**	
Apical third	Middle third	Apical third	Middle third	Apical third	Middle third	Apical third	Middle third	Apical third	Middle third
(5.5%)	(5.5%)	(5.5%)	0	(11.1%)	(11.1%)	0	0	0	0

Table 3. The presence of corrosion on the sets Ni-Ti instruments after instrumentation, disinfection and sterilization

Ni-Ti instruments	I group		II group		III group	
K3	100%	100%	100%	100%	100%	100%
Mtwo	16.7%	16.7%	33.3%	50%	0	16.7%
ProTaper Universal	100%	100%	100%	100%	100%	100%
HyFlex	100%	100%	6.7%	16.7%	83.3%	66.7%
BioRaCe	0	0	0	0	0	0



A B

Figure 1. Spin echo image of the new Mtwo instrument (10 / 0.4): A – surface of the middle third on which the presence of corrosion is observed (× 170); B – detail from the previous image at higher magnification (× 600)

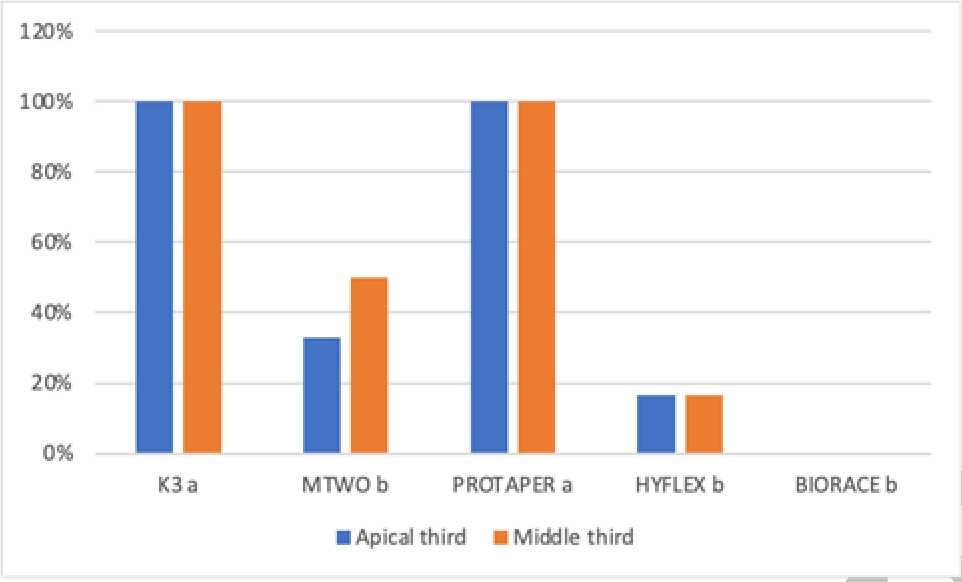


Figure 2. Prevalence of corrosion on Ni-Ti instruments after instrumentation of disinfection and sterilization in the canals of the first group; similar letters (a or b) indicate no statistically differences ($p > 0.05$)

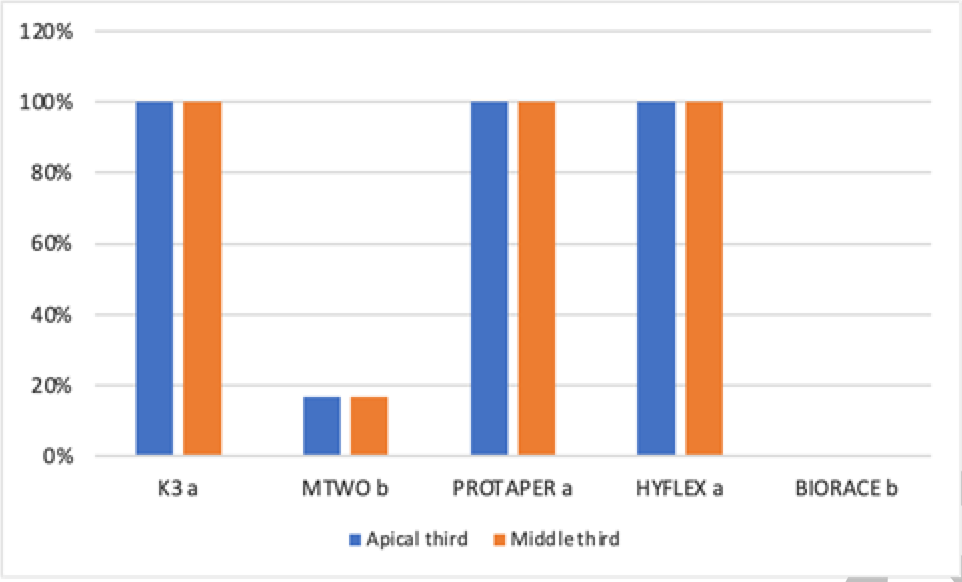


Figure 3. Corrosion prevalence on Ni-Ti instruments after instrumentation, disinfection and sterilization in the channels of the second group; similar letters (a or b) indicate no statistically differences ($p > 0.05$)

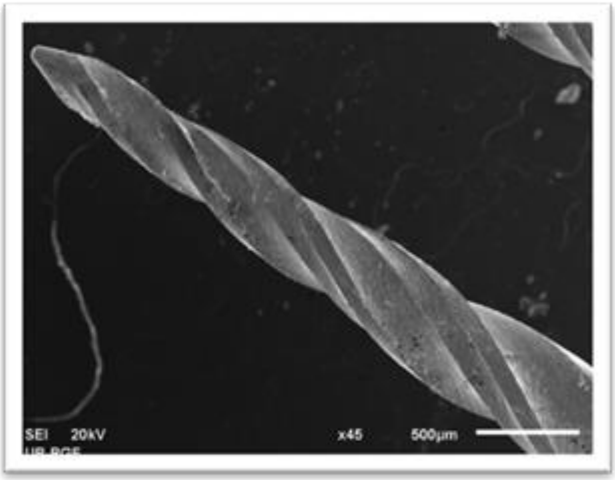


Figure 4. SE image K3 instrument (25–0.08), second experimental group (corrosion on the apical third) (× 45)

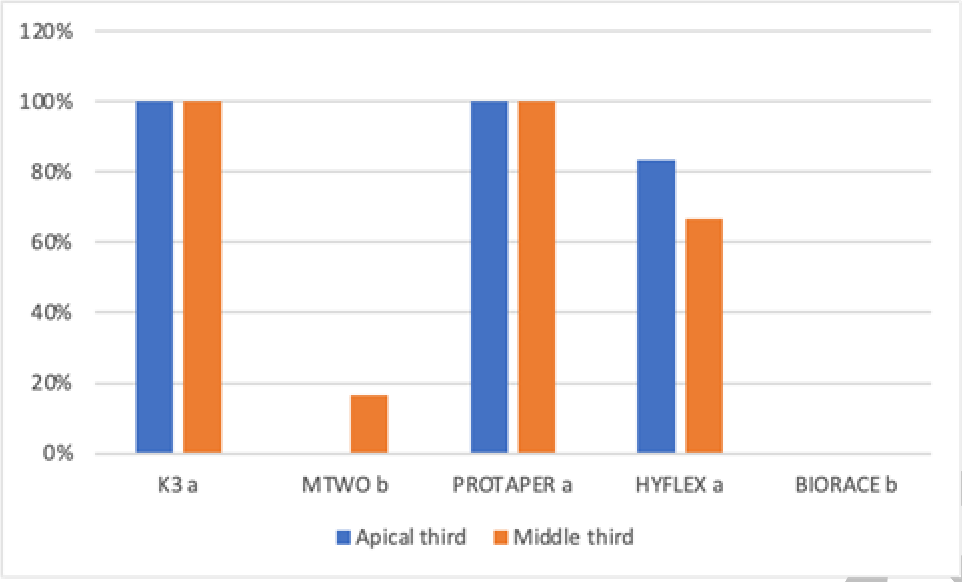


Figure 5. Prevalence of corrosion by Ni-Ti instruments after instrumentation, disinfection and sterilization of third group; similar letters (a or b) indicate no statistically differences ($p > 0.05$)

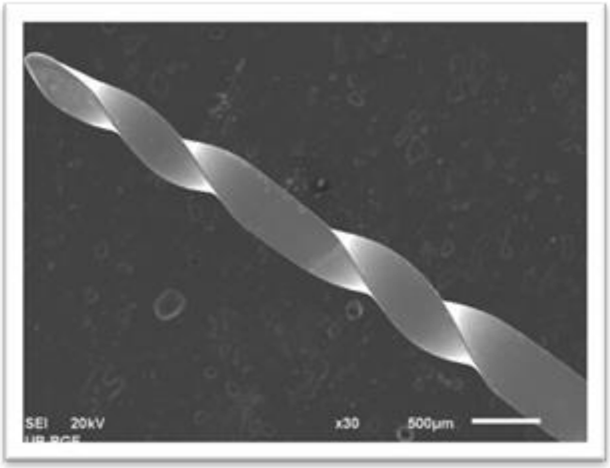


Figure 6. Spin echo image BioRaCe instrument no 4 without incorrection and corrosion (× 30)