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ORIGINAL ARTICLE / ОРИГИНАЛНИ РАД

Analysis of working surface in new manual and rotary endodontic instruments (scanning electron microscopy)

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SUMMARY

Introduction/Objective The objective of this study is to use scanning electron microscopy (SEM) to analyze working surfaces of new manual and rotary endodontic instruments and to check possible existence of manufacture dirt particles or defects on the working surface.

Methods In this study, we used three sets of new manual instruments: K-File, KF (Dentsply Maillefer, Switzerland) and Hedstorm Files, HF (SybronEndo Co, USA) and three sets of mechanical Ni-Ti instruments – type K3 (SybronEndo Co, USA) and BioRaCe (FKG DENTAIRE Swiss Dental Products, Switzerland). The instruments were analyzed using SEM method at 170 × magnification while semi-quantitative energy dispersive x-ray analysis was used to determine chemical composition of dirt particles. Fisher test ($p < 0.05$) was applied in statistical analysis.

Results Results showed that none of the instruments were defect-free. The most common defect type was the presence of metal strips, which were noticed at the surface of all tested instruments. Debris was present on all manual and only one type of mechanical instruments, K3 (39% in the apical and 33% in the middle third). Fretting was noticed in all manual KF and all mechanical instruments of the K3 group. Pitting was common in all manual instruments, KF (33% in the apical and 39% in the middle third) and HF (11% in the apical and 6% in the middle third). Corrosion of the working surface, metal flash, and disruption of the cutting edge were marked only in the KF group.

Conclusion Manufacture defects were noticed in all instruments and the most common type of irregularity were metal strips. Electropolished surface of BioRaCe instruments showed no debris of organic origin.

Keywords: stainless-steel manual instruments; Ni-Ti rotary instruments; defects; SEM; debris

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INTRODUCTION

Clinical endodontics implies the so-called “cleaning and shaping” concept, with cleaning the complex endodontic space from vital, necrotic, or infected pulp tissue, bacteria and their products and shaping while preserving the original form of the radix canal [1, 2, 3].

Chemomechanical root canal treatment is usually performed with manual endodontic instruments (made of stainless steel or Ni-Ti alloy) or mechanical Ni-Ti rotary endodontic instruments with adequate and abundant irrigation of the canal system. The use of rotary Ni-Ti files in endodontic practice reduces the possibility of errors during instrumentation, such as obstruction, steps, transportation, and perforation of the canal wall [4].

Even though Ni-Ti rotary instruments are more efficient when compared to manual in almost every aspect (speed, simplicity, and uniformity of instrumentation), their drawback is the possibility of deformation and fracture during instrumentation, most likely due to inadequate use [5].

A fractured instrument is a serious threat to treatment, irrigation, and filling of root canals and it may significantly affect the outcome of endodontic treatment [6]. Numerous studies researched the factors which can influence deformation and fracture of manual and mechanical endodontic instruments: Parashos et al. [7], Di Fiore [8], Shen et al. [9], Kosti et al. [10], Priyanka et al. [11], Gil et al. [12]. In 2018, Boutsioukis and Lambrianidis [13] made an outline of all these factors and grouped them into four categories: operator-related factors, anatomy-related factors, instrument-related factors and technique/use-related factors.

It is confirmed that endodontic instruments, due to their design and different manufacture process, may significantly impact deformation and fracture during root canal instrumentation [10]. Most of new endodontic instruments are non-sterile and can contain various metal debris, dirt particles, and epithelial cells on their surface. The process of production of stainless steel files can lead to the presence of small metal scraps that are more or less retained at the work surfaces of the files [14].

Stainless steel endodontic instruments are usually made by twisting of various steel profiles by longitudinal axis, thus forming blades from vertical wire edges [15]. Ni-Ti rotary instruments are used in endodontic procedure due to their good mechanical properties, biocompatibility, ductility, corrosion resistance, low elastic modulus, and special characteristics such as super-elasticity and shape memory effect [16]. Production of Ni-Ti mechanical endodontic instruments is more complex when compared to the process of steel instrument manufacture. Due to memory property of the Ni-Ti alloy, the majority of instruments are manufactured by engraving on a milling machine rather than by twisting [15]. Despite the fact that top-notch computer technology is used to manufacture very complicated Ni-Ti instruments (computer-aided design and computer-aided manufacturing – CAD-CAM), surface defects including fretting, cracks, pitting, and dirt are very common [15]. Irregularities at the instrument surface might increase its vulnerability to fracture. Surface defects seem to be points of tension and can cause initiation and spread of cracks, thus potentially highly contributing to possible fractures during instrument activation [13].

The aim of this study is to use a scanning electron microscope (SEM) to analyze working surfaces of new manual and mechanical endodontic instruments and to check possible existence of manufacture impurities or defects on the working surface.

METHODS

This research implied the use of three basic sets (each set consisting of six instruments) of new manual stainless steel instruments: K-File – KF (Dentsply Maillefer, Ballaigues, Switzerland) and Hedstorm Files – HF (SybronEndo Co, Orange, CA, USA) and three basic sets (each set consisting of six instruments) of machine endodontic instruments, type K3 (SybronEndo Co) and BioRaCe (FKG DENTAIRE Swiss Dental Products, La Chaux-de-Fonds, Switzerland). SEM analysis (JEOL JSM-6610LV, Tokyo, Japan) was carried out at the SEM-EDS (energy dispersive X-ray spectroscopy) laboratory of the Faculty of Mining and Geology, University of Belgrade, without any prior preparation.

Microphotographs are realized at 170 × magnification, but in case of noticeable changes on the instruments, for the purpose of more detailed analysis, they are carried out at magnification of up to 800 ×. The apex and the middle third of the instruments were analyzed from two different directions and each side of instrument was analyzed by three images.

Analysis of different irregularities and omissions during manufacture process implied the criteria proposed by Eggert et al. [17]: score 1 – no visible defect; score 2 – pitting; score 3 – fretting; score 4 – micro fractures; score 5 – complete fracture; score 6 – metal flash; score 7 – metal strips; score 8 – blunt cutting edge; score 9 – disruption of cutting edge; score 10 – corrosion; score 11 – debris. Qualitative analysis was performed, though obtained results were not quantified. Semi-quantitative Energy Dispersive X-Ray Analysis (EDXS) determined chemical composition of the found dirt particles.

The study was approved by the Ethics Commission of the School of Dental Medicine, University of Belgrade (36/6).

Statistical analysis of obtained results was performed using the Fisher's test ($p < 0.05$).

RESULTS

Obtained results are presented in Tables 1 and 2 and Figures 1–9.

The results showed that all tested instruments had some kind of defect on their working surface. New manual and mechanical instruments did not show any signs of micro fractures, fractures, or blunt cutting edges (Table1).

Metal strips were the most common defect type noticed on the surface of all tested instruments (Figure 1).

In K3 rotary group of Ni-Ti instruments, the presence of this contamination was 89% in the apical and 78% in the middle third. In all other groups, this defect was present in 100% of the cases (Table 1, Figure 2). Fisher's test statistical analysis did not show any significant differences between the tested instruments and their apical or middle thirds.

Analysis of SEM microphotographs determined the contamination of the working surfaces of the tested instruments, and subsequent EDXS defined the chemical composition of the contamination (Figure 3, Table 2). Thus, we divided instruments into two types – instruments contaminated

Table 1. Presence of defects and dirt on the working surface of the tested instruments

Defects	KF		HF		K3		BioRaCe	
	Apical third	Middle third	Apical third	Middle third	Apical third	Middle third	Apical third	Middle third
Pitting	6	7	2	1	/	/	/	/
Fretting	18	18	/	/	18	18	/	/
Metal-flash	2	1	/	/	/	/	/	/
Metal-strips	18	18	18	18	16	14	18	18
Disruption of cutting edge	1	/	/	/	/	/	/	/
Corrosion	2	3	/	/	1	1	/	/
Debris	18	18	10	10	7	6	/	/

HF – Hedstorm files; KF – K-file

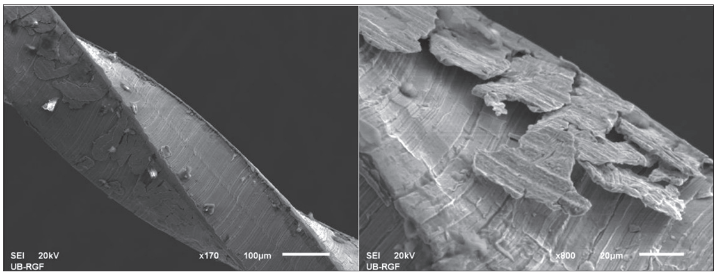

Figure 1. (a) Scanning electron microscopy analysis of a K-File instrument working surface (middle third) with metal strips and fretting (magnification 170 ×); (b) detail from picture (a), metal strips on working surface of the K-File instrument (magnification 800 ×)

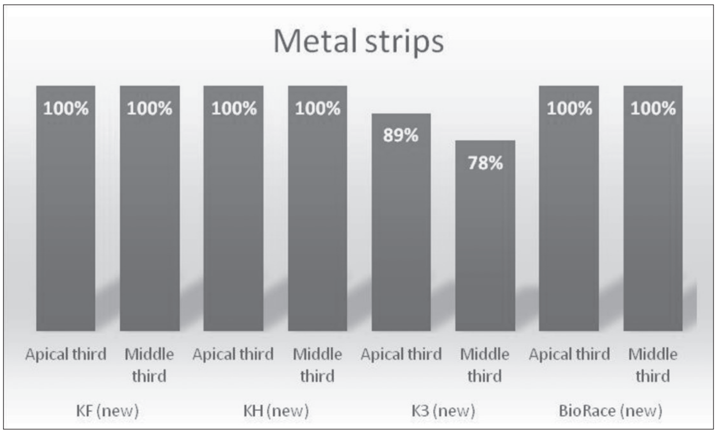

Figure 2. The presence of metal strips on the working surface of the tested instruments

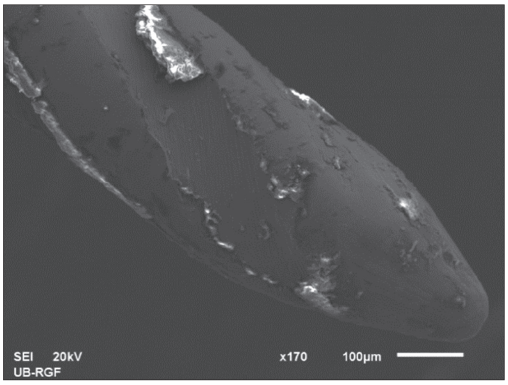

Figure 5. Scanning electron microscopy analysis of the working surface of K3 instruments (apical third) with metal strips, fretting, and debris (magnification 170 ×)

Table 2. Chemical composition of impurities (spectrums 1 and 2) and clean K-3instrument (spectrum 3); chemical analyses are given in wt.% and are normalized to 100 wt.%

Spectrum	C	N	O	Na	Mg	Al	Si	S	Cl	K	Ca	Ti	Fe	Ni	Total
1	47.7	0	37.4	2.3	1	0.2	2.5	0.9	3.6	1.6	1.1	0.6	0	1.1	100
2	49.9	0	34.1	0.7	0.6	0.4	1.8	0.3	0.5	0.4	7.6	2	0	1.7	100
3	0	0	0	0	0	0	0	0	0	0	0	44.3	0	55.7	100

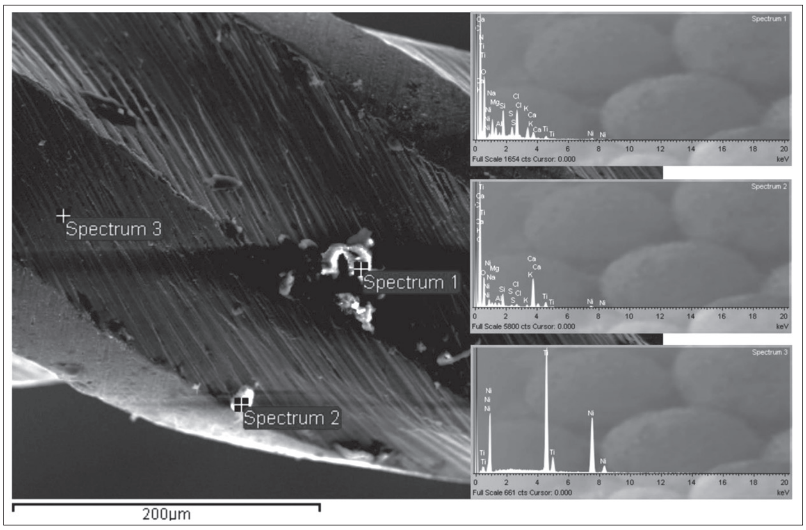

Figure 3. Energy-dispersive X-ray spectroscopy analysis

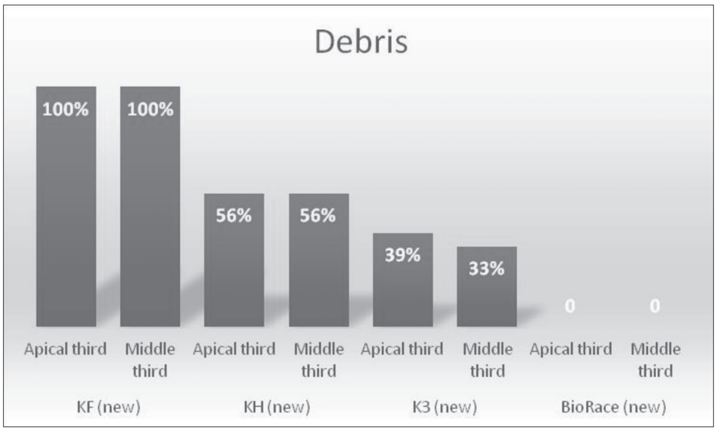

Figure 4. The presence of debris on the working surface of the tested instruments

with metal strips and instruments with debris.

Debris was present in all manual instruments: KF instruments (100% in the apical and middle third) and HF instruments (56% in the apical and middle third). This contamination type was marked in only one type of mechanical Ni-Ti instruments – K3 (39% in the apical and 33% in the middle third), while BioRaCe group did not show any presence of debris. (Table 1, Figure 4). While comparing debris in different manual instruments (KF and HF), a statistically significant difference was noticed ($p = 0.0029$ in the apical and $p = 0.0029$ in the middle third). Also, among different mechanical Ni-Ti instruments (K3 and BioRaCE), there was a statistically significant difference in debris contamination; ($p = 0.076$ in the apical and $p = 0.0191$ in the middle third). Statistically significant difference was also noticed when all manual (KF and KH) instruments were compared to all mechanical rotary Ni-Ti (K3 and BioRaCe) instruments: ($p = 0.0001$in the apical and $p = 0.0001$ in the middle third).

Fretting caused by manufacture was noticed in all manual instruments of the KF group and all mechanical Ni-Ti instruments of the K3 group, while manual HF and mechanical BioRaCe instruments did not show any signs of fretting (Table 1, Figure 5). While comparing fretting on different manual instruments (KF and HF), a statistically significant difference was noticed in the apical and the middle third ($p = 0.0001$ in the apical and $p = 0.0001$ in the middle third). In addition, among mechanical Ni-Ti instruments (K3 and BioRaCE), there was a significant statistical difference in fretting on the working surface ($p = 0.0001$ in apical and $p = 0.0001$ in middle third). A statistically significant difference in

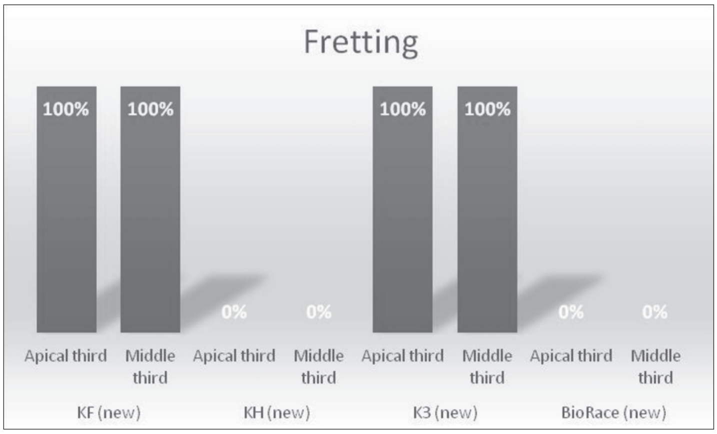

Figure 6. The presence of fretting on the working surface of tested instruments

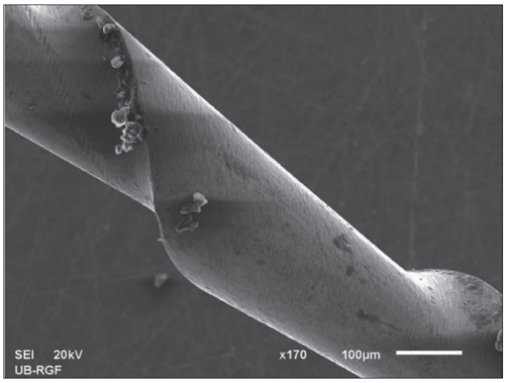

Figure 8. Scanning electron microscopy analysis of the working surface of a Hedstorm file instrument (middle third) with pitting and debris (magnification 170 ×)

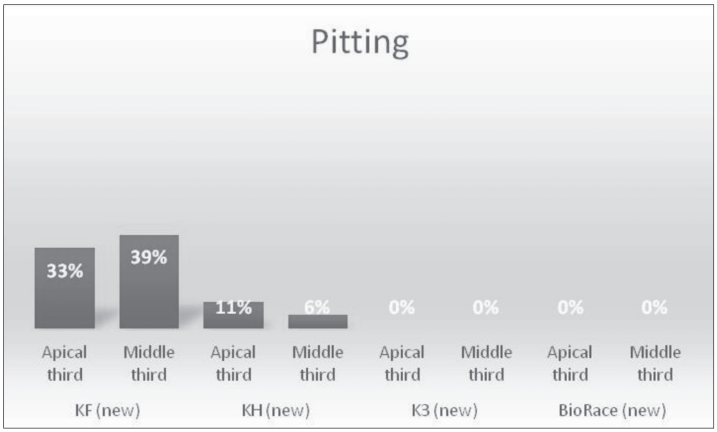

Figure 7. The presence of pitting on the working surface of the tested instruments

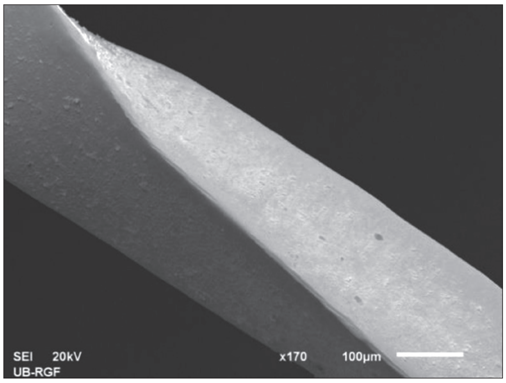

Figure 9. Scanning electron microscopy analysis of theworking surface of a BioRaCe instrument (middle third) with a small number of metal strips on the electropolished surface (magnification 170 ×)

fretting was not marked when all manual instruments (KF and HF) were compared to all mechanical rotary Ni-Ti (K3 and BioRaCe) instruments (Figure 6).

Pitting was marked in the apical and the middle third of the manual instruments, KF (33% in the apical and 39% in the middle third) and HF (11% in the apical and 6% in the middle third), though none of the groups of Ni-Ti instruments (Figure 4) showed any signs of pitting (Table 1, Figures 7 and 8). While comparing pitting in various manual instruments (KF and HF), a statistically significant difference was noticed in the apical third ($p = 0.0051$), which was also marked in the middle third ($p = 0.0045$). Among tested mechanical Ni-Ti instruments, there was no statistically significant difference in pitting (K3 and BioRaCE). Statistically significant difference in pitting was noticed by mutual comparison of all manual (KF and HF) and all mechanical rotary (K3 and BioRaCe) instruments ($p = 0.0051$ in the apical and $p = 0.0051$ in the middle third).

Corrosion of the working surface, metal flash, and disruption of cutting edge were marked only in KF instruments (corrosion – 11% in the apical and 17% in the middle third; metal flash – 11% in the apical and 6% in the middle third; disruption of cutting edge – 2% in the apical third) (Table 1) In the second group of KH manual instruments and in both groups of mechanical NI-Ti instruments (K3 and BioRaCe), corrosion, metal flash, and disruption of the cutting edge were not detected (Figure 9).

DISCUSSION

Above all, success of endodontic therapy depends on proper instrumentation and biomechanical cleaning of the root canal. Design of endodontic instruments, their metallurgical characteristics and surface may complicate endodontic treatment in case the instrument deforms or fractures during use. It is proved that manufacture defects might cause fracture of new instruments even during their first clinical use [18].

Results of this study showed that all analyzed instruments had at least one (or more) different defects prior to any use. Defects found on new instruments only confirm the difficulties in manufacture of specific endodontic instruments, and issues that may arise during their clinical use. New processes of instruments' manufacture that aim at improving used materials, minimizing inherent defects and increasing the resistance of instruments to deformation and fracture very often remain undisclosed, usually due to manufacturers' originality and patents [19].

Fretting caused by manufacture process (due to milling), was marked in almost all tested manual steel instruments. Manufacture process of rotary Ni-Ti endodontic instruments is much more complex than the process of stainless steel instruments manufacture since they have to be mechanically treated instead of just being twisted as steel instruments. As a result, the problem is in this case even more significant.

Attempts to conventionally manufacture Ni-Ti instruments by twisting the wire would probably result in their fracture; therefore, shaping of these instruments is achieved by even pressure from a series of rollers applied to the Ni-Ti wire, thus defining its shape, conicity, form of blade edges, i.e. design characteristics of such instruments. Difficulties during manufacture of rotary endodontic instruments and elimination of surface irregularities such as traces of milling and metal flash (especially on blades) which might compromise efficiency of instrument blade and potentially cause problems related to corrosion or fracture [18].

Presence of metal strips, noticed in all the tested groups with the score of 100% apart from group K3 (78% in the middle and 89% in the apical third), confirms the complexity of endodontic instrument manufacture.

Pitting on the working surface of an instrument was noticed only in case of manual steel instruments (KF, HF), and it could be explained by the specific technological process of manufacture, as is the case with the presence of metal flash and defects on the cutting edge in the KF group.

Manufacturers are constantly in search of metallurgical modifications of the Ni-Ti alloy, trying to find a perfect solution that would increase the performance in terms of super-elasticity and memory properties of the alloy [20]. During the manufacturing process, problems might occur due to the quality of the Ni-Ti alloy since particles of oxide might stay incorporated in the alloy during the manufacturing process. Instrumentation and stress propagation might impact these points and make nucleation spots for the development of micro-fractures and possible instrument defects [21].

Composition of the Ni-Ti alloy used for the construction of endodontic instruments is 56% (weight) of nickel and 44% (weight) of titanium, thus achieving their equiatomic relation. Despite the fact that only one manufacturer (Dentsply Maillefer Instruments) has revealed the complete composition and detailed technological process of rotary instruments manufacture, it is assumed to be the only relation between the elements that gives alloy its super-elastic characteristic. Variation of the Ni-Ti alloy composition enables the manufacture of instruments with different properties: either super-elastic alloy or better memory property. Increasing the percentage of nickel or replacing the elements in traces (e.g. cobalt) results in decreased temperature of transformation. Increase in annealing temperature increases the transformation temperature as well [18].

Results of this study showed the presence of debris in all instruments apart from the BioRaCe group of instruments. This is explained by the existence of electrochemically treated working surface of BioRaCe instruments, thus achieving better cutting edge efficacy and resistance to wear and tear. Electrochemical polishing decreases irregularities at the surface of instruments, thus decreasing the accumulation of organic debris [22, 23].

In order to decrease surface defects and improve resistance of endodontic instruments, various methods are applied: alloy implantation process by argon, boron, or nitrogen ions, thermal nitridation (coating of instruments with a layer of titanium nitride), plasma immersion, deep dry cryogenic treatment and electro polishing [20, 22–25]. Many researchers were focused on increasing efficiency and flexibility and decreasing resistance to cyclic fatigue. As a result, they suggest additional thermo-mechanical treatment of the raw Ni-Ti alloy or even finished instruments [25, 26, 27]. The process of vapor accumulation enables treatment of Ni-Ti instruments with a layer of titanium nitride, thus achieving better cutting-edge efficiency and resistance to corrosion [27]. Memory-shape alloys based on the equiatomic Ni-Ti composition have great importance in the development of modern endodontic rotary instruments [16]. It is necessary and very important to be familiar with metallurgical characteristics of the Ni-Ti alloy in order to use their clinical potential in the best possible way and minimize frustration and fear that many dentists have that such instruments could fracture during root canal treatment [28, 29].

CONCLUSION

Based on the results of this research, it is concluded that all tested instruments showed manufacture defects (one or more), and that the most common defect type was the presence of metal strips on working surfaces of instruments. Due to the presence of debris on working surfaces of instruments, it is necessary to sterilize the instruments before initial use. Electropolished surfaces of BioRaCe instruments showed no presence of organic debris. These facts could be a warning sign to all practitioners – before initial use, the working surface of instruments should be well analyzed in order to avoid possible complications during endodontic treatment.

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Анализа површине радног дела нових ручних и машинских ендодонтских инструмената (скенирајућа електронска микроскопија)

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САЖЕТАК

Увод/Циљ Циљ овог рада је био да се применом скенирајуће електронске микроскопије (СЕМ) анализирају површине нових ручних и машинских ендодонтских инструмената и провери евентуално постојање производних нечистоћа или дефеката на радном делу.

Методе У истраживању су коришћена по три сета нових ручних инструмената: *K-File*, *KF* (*Dentsply Maillefer*, Швајцарска) и *Hedstorm Files*, *HF* (*SybronEndo Co*, САД) и по три сета машинских *Ni-Ti* инструмената, типа К3 (*SybronEndo Co*) и *BioRaCe* (*FKG DENTAIRE Swiss Dental Products*, Швајцарска). Инструменти су подвргнути СЕМ анализи са увећањем 170×, а семиквантитативном, *EDXS* анализом утврђиван је хемијски састав нечистоће. Статистичка анализа је урађена применом Фишеровог теста ($p < 0,05$).

Резултати Резултати су показали да не постоји ниједан инструмент без дефекта. Најучесталији тип дефекта је било присуство металних опиљака, који су уочени на површини свих испитиваних инструмената. Дебри је уочен на свим ручним и само на једном типу машинских инструмената, К3 (39% на апикалној и 33% на средњој трећини). Жлебови су уочени на свим ручним *KF* и свим машинским инструментима групе К3. Присуство удубљења забележено је код ручних инструмената, *KF* (33% апикална и 39% средња трећина) и *HF* (11% апикална и 6% средња трећина). Корозија радне површине, појава углачане површине и прекид сечивне ивице су уочени само у групи *KF*.

Закључак На свим испитиваним инструментима су уочени производни дефекти, а најучесталији тип неправилности су метални опиљци. На електрополираној површини инструмената *BioRaCe* није уочено присуство органског дебрија.

Кључне речи: челични ручни инструменти, *Ni-Ti* ротирајући инструменти, дефекти, СЕМ, дебри