

Local Elements Content on Working Surfaces of Five Different New Ni-Ti Rotary Endodontic Files: SEM-EDS Study

Igor Noenko^{1,*}, Volodymyr Fedak² and Anastasia Biley³

¹National Healthcare University of Ukraine, All-Ukrainian Association of Endodontists, Kyiv, Ukraine

²Private practice, All-Ukrainian Association of Endodontists, Chernivtsi, Ukraine

³Uzhhorod National University, Uzhhorod, Ukraine

Abstract: *Objective:* To assess diversity of local elements content within working surfaces of five different new Ni-Ti rotary endodontic files.

Methods: Energy dispersive spectrometry (EDS) for local elements content analysis was held within three specific areas of working surfaces of the files identified over received SEM images: 1) with pronounced visible signs of impurities; 2) with slightly visible signs of impurities; 3) with no visible signs of impurities. EDS procedure was provided with the use of EDS-detector. Local elements content data at the three above-mentioned specific areas of files' working surfaces was presented in means of weight percent (wt%), normalized to 100%, and was automatically calculated by the SEM-EDS software.

Results: Expressiveness of visible signs of impurities present over working surfaces of the files demonstrated correlation with nickel ($r=-0.62$, $p < 0.05$) and titanium ($r=-0.49$, $p < 0.05$) content at analyzed areas, while also with carbon ($r=0.57$, $p < 0.05$) and oxygen ($r=0.50$, $p < 0.05$) content at analyzed areas. Odds ratio for possibility to register traces of one of the following elements N, Na, Mg, Al, Si, S, Cl, Cr, Fe, Ca at the working surface of endodontic file under condition of present pronounced visible signs of impurities was found to be at the level of OR=12.5 (95% CI: 1.08; 143.44; $p=0.04$).

Conclusions: None of the analyzed files was free of impurities over their working surface. Carbon, oxygen, nickel, and titanium were the most prevalent elements observed along working surfaces of five new different endodontic files, while some instruments were characterized with the presence of tungsten, silicon, aurum, iron, chromium, magnesium and nitrogen, maximum level of which at selected areas with pronounced visible signs of impurities outreach 10% weight percent values.

Keywords: Heat treatment, NiTi instruments, Elemental analysis, Endodontic files, Flexibility, Microstructure.

INTRODUCTION

Ni-Ti rotary instruments have changed workflow of endodontic treatment by optimizing clinical process, decreasing number of needed files and improving outcome of interventions provided within root canals [1-3]. Nowadays development of Ni-Ti rotary files has not stopped and still ongoing, while including improvements within their mechanical and metallurgical characteristics [4]. Gambarini *et al.* highlighted that novel strategy of Ni-Ti files development focused over improved approaches of their thermomechanical processing [5]. On the other hand continuous development and improvements of Ni-Ti files requires adapted approaches for their quality assurance, considering specific features of instruments manufacturing [6-8].

Scanning electron microscopy (SEM) combined with energy dispersive spectrometry (EDS) have been widely used as non-destructive methods in number of

studies provided for the assessment of endodontic files' surface [6, 7, 9]. Based on the obtained outcomes it was concluded that even new endodontic instruments may demonstrate various surface defects, most of which were related with the specific process of their manufacturing and packaging [6, 11-14]. On the other hand SEM-EDS approach also helped to revealed that counterfeit endodontic rotary instruments characterized with increased number of different surface defects, while also with prominent SEM-differences compared to original design of the file [7].

Even though SEM-EDS approach allows to provide elements content analysis of endodontic files' surface, only limited amount of studies have been focused on such specific aspect [7, 12-14]. Previously it was demonstrated that presence of carbon and oxygen within rotary instrument may affect its' structural integrity and rigidity thus influencing its' performance, but till now no clear conclusions have been made regarding the impact of different chemical elements accumulation on the functional prognosis of Ni-Ti instruments [15]. It is known that traces of some chemical elements other than nickel and titanium may

*Address correspondence to this author at the National Healthcare University of Ukraine, All-Ukrainian Association of Endodontists, Kyiv, Ukraine; E-mail: bban@i.ua

be found over rotary file working surfaces [13], but it is not clarified accumulation of such to which extent may be interpreted as clinically insignificant. On the other hand data obtained both during SEM analysis, while also during EDS analysis potentially may be used as evidences for Ni-Ti files fabrication and packaging quality assurance [6].

Considering all above mentioned facts it seems relevant to provide research dedicated to the analysis of local elements content over the different areas of endodontic files' working surfaces with various levels of visible impurities signs, while also to compare such parameters among files of different brands, considering differences in their manufacturing process due to the patented technologies.

OBJECTIVE

To assess diversity of local elements content within working surfaces of five different new Ni-Ti rotary endodontic files.

MATERIALS AND METHODS

Study Sample

Endodontic instruments in amount of 10 pieces of five different brands (SANI for retreatment (Chengdu Sani Medical Equipment Co, Chengdu, China) – size 25, taper 2%; Soco SC Plus (Chengdu Sani Medical Equipment Co, Chengdu, China) – size 20, taper 4%; HyFlex CM (Coltene, Altstätten, Switzerland) – size 20, taper 4%; Profile (Dentsply Sirona, Salzburg, Austria.) – size 20, taper 4%; PreRace (FKG Dentaire, La Chaux-de-Fonds, Switzerland) – size 30, taper 6%) were bought from official distributors in Ukraine, thus forming primary cohort of 50 endodontic files. Out of 50 available instruments one piece of each brand was randomly selected for further scanning electron microscopy procedure and subsequent local elements content analysis.

Scanning Electron Microscopy (SEM) and Energy Dispersive Spectrometry (EDS) Analysis

Scanning electron microscopy of the endodontic files was provided within Laboratory of Electron Microscopy «Nano Technologies in Medicine» (Kyiv, Ukraine) [16]. Randomly selected file of each brand was carefully removed from the original packaging, and transfer to the holder of scanning electron microscope (Tescan Mira3 LMU, TESCAN, Brno, Czech Republic). File transfer from original packaging to the holder within

microscope was provided without any contact with working surfaces of the files. No specific preparation or processing of the files was held before procedure of scanning electron microscopy. Scanning electron microscope was tuned with the following parameters for the aim of present research: SEM HV (acceleration voltage) – 10.0 kV, SEM MAG (magnification) ranging from 23× to 379×, view field ranging from 300 μm to 5.0 mm, with use of SE (secondary electrons) and BSE (backscattered electrons) detectors.

Energy dispersive spectrometry (EDS) for local elements content analysis was held within three specific areas of working surfaces of the files identified over received SEM images: 1) with pronounced visible signs of impurities; 2) with slightly visible signs of impurities; 3) with no visible signs of impurities. EDS procedure was provided with the use of EDS-detector (Oxford X-max 80 mm, Oxford Instruments, Abingdon, UK), mounted within the structure of scanning electron microscope. Local element content data at the three above-mentioned specific areas of files' working surface was presented in means of weight percent (wt%), normalized to 100%, and was automatically calculated by the SEM-EDS algorithm within TESCAN Essence software (TESCAN, Brno, Czech Republic).

Statistical Analysis

Obtained EDS results regarding local elements content were systematized within Microsoft Excel software version 16.0 (Microsoft Office 2019, Microsoft Corporation India Pvt. Ltd., New Delhi, India), and grouped for Shapiro-Wilk testing with the aim to establish adherence of received data to the normal distribution pattern [17]. ANOVA analysis was provided for the data with normal distribution. Fisher's criterion ($p < 0.05$) was applied during the processing of results obtained after EDS-analysis for establishing the significance of differences regarding content of different elements observed at areas with various visible signs of impurities along working surface of endodontic file. Weight percentage (wt%) of each identified element within three different areas was presented in the form of normalized value considering 100% margin of overall content.

Ethical Aspects

Considering that present research represents laboratorial analysis of the endodontic instruments without participation of any human or animal subjects the ethical approval for such was not needed, however

taking onto account that present study is a part of comprehensive investigation project, complex design of latter and its adherence to all relevant ethical norms was ratified by Institutional Review Board of Faculty of Dentistry at Uzhhorod National University (Ukraine). Comprehensive scientific research project, which includes present study as a component part, realized by the Department of Restorative Dentistry at Uzhhorod National University (Ukraine), and aimed at the implementation of modern materials and technologies into the dental practice with further assessment of their effectiveness.

RESULTS

SEM-EDS analysis of SANI for retreatment file revealed that areas with no visible signs of impurities (Spectrum 2 on Figure 1) consist of 54.56% of nickel, 37.39% of titanium, 5.85% of carbon and 2.20% of oxygen. Area with slightly visible signs of impurities (Spectrum 1 on Figure 1) consists of lower amounts of nickel and titanium, which were not statistically differed

from such registered at Spectrum 2 ($p > 0.05$); however wolfram was observed in statistically significant amounts in Spectrum 1 compared to Spectrum 2 ($p < 0.05$). Carbon and oxygen amounts in Spectrum 1 were analogical to such at Spectrum 2 ($p > 0.05$). Spectrum 3 data, which was registered at the area with pronounced visible signs of impurities, demonstrated significant increase of carbon and oxygen ($p < 0.05$), while also in parallel amounts of nickel and titanium were significantly decreased ($p < 0.05$). EDS analysis of area with pronounced visible signs of impurities also revealed traces of silicon, aluminum, calcium and sulfur (Table 1).

Soco SC Plus file's working surface demonstrated presence of 57.20% of nickel and 36% of titanium with minimal amounts of carbon and oxygen at the area with no visible signs of impurities (Spectrum 1 on Figure 2). Spectrum 3 was used for marking area with slightly visible signs of impurities over SEM image of working surface for above mentioned file, and such was characterized with statistically insignificant

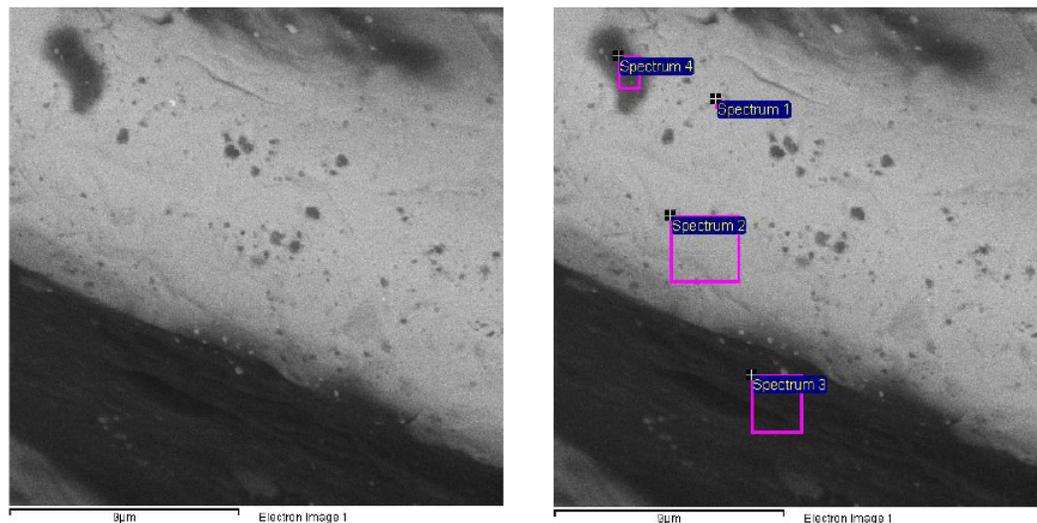


Figure 1: SEM image of SANI for retreatment file's working surface with markings of areas, which were analyzed for local element content through EDS (Spectrum 1 – area with slightly visible signs of impurities, Spectrum 2 – area with no visible signs of impurities, Spectrum 3 – area with pronounced visible signs of impurities).

Table 1: Local Elements Content Registered Through EDS Among Areas with Different Visible Signs of Impurities along Working Surface of SANI for Retreatment File

| Spectrum | C (wt%) | O (wt%) | Al (wt%) | Si (wt%) | S (wt%) | Ca (wt%) | Ti (wt%) | Ni (wt%) | W (wt%) | Total (wt%) |
|------------|---------|---------|----------|----------|---------|----------|----------|----------|---------|-------------|
| Spectrum 1 | 5.33 | 4.35 | 0.38 | - | - | - | 26.94 | 38.83 | 24.17 | 100.00 |
| Spectrum 2 | 5.85 | 2.20 | - | - | - | - | 37.39 | 54.56 | - | 100.00 |
| Spectrum 3 | 70.99 | 22.09 | 1.37 | 1.87 | 0.23 | 0.41 | 1.51 | 1.52 | - | 100.00 |

Note: Spectrum 1 – area with slightly visible signs of impurities, Spectrum 2 – area with no visible signs of impurities, Spectrum 3 – area with pronounced visual signs of impurities.

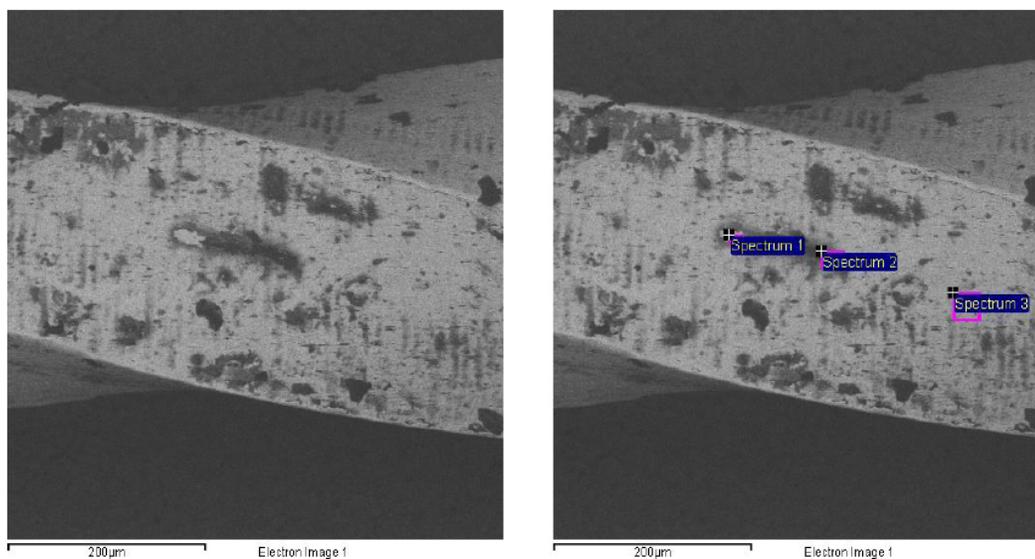


Figure 2: SEM image of Soco SC Plus file's working surface with markings of areas, which were analyzed for local element content through EDS (Spectrum 1 – area with no visible signs of impurities, Spectrum 2 – area with pronounced visible signs of impurities, Spectrum 3 – area with slightly visible signs of impurities).

Table 2: Local Element Content Registered Through EDS among Areas with Different Visible Signs of Impurities Along Working Surface of Soco SC Plus File

| Spectrum | C (wt%) | N (wt%) | O (wt%) | F (wt%) | Na (wt%) | Mg (wt%) | Si (wt%) | Cl (wt%) | Ca (wt%) | Ti (wt%) | Ni (wt%) | Total (wt%) |
|------------|---------|---------|---------|---------|----------|----------|----------|----------|----------|----------|----------|-------------|
| Spectrum 1 | 5.25 | 0.00 | 1.55 | | | | | | | 36.00 | 57.20 | 100.00 |
| Spectrum 2 | 43.03 | | 34.20 | 0.93 | 0.62 | 0.28 | 1.84 | 1.30 | 1.10 | 9.49 | 7.21 | 100.00 |
| Spectrum 3 | 8.15 | | 2.69 | | | | | | | 34.88 | 54.28 | 100.00 |

Note: Spectrum 1 – area with no visible signs of impurities, Spectrum 2 – area with pronounced visible signs of impurities, Spectrum 3 – area with slightly visible signs of impurities.

decrease of nickel and titanium ($p > 0.05$), while also with minimum increase of carbon and oxygen amounts ($p > 0.05$). Area with pronounced visible signs of impurities (Spectrum 2) represented decrease of both titanium and nickel ($p < 0.05$), while also sufficient changes in amounts of carbon and oxygen ($p < 0.05$), and minimal traces of fluorine, sodium, magnesium, silicon, chlorine, and calcium (Table 2).

EDS of HyFlex CM endodontic file's working surface revealed that area with no visible signs of impurities (Spectrum 1 on Figure 3) was characterized with 32.56% of titanium, 24.88% of nickel, 26.67% of aurum, 4.07% of carbon, 10.95% of oxygen, while also traces of aluminum and silicon were found in amounts of 0.35% and 0.52% respectively. Area with slightly visible signs of impurities (Spectrum 2 on Figure 3) were characterized with significant increasing changes of carbon and oxygen ($p < 0.05$), while also decreasing amounts of titanium and nickel ($p < 0.05$), and full

absence of aurum ($p < 0.05$). Changes in area with pronounced visible impurities (Spectrum 3) demonstrated analogical pattern to above-mentioned, but such was more expressive in absolute values, while also traces of sodium, magnesium, aluminum, silicon, sulfur, potassium, calcium and chlorine were noted (Table 3).

Profile demonstrated presence of 37.93% of titanium, 53.42% of nickel, 8.29% of carbon and 0.36% of sulfur within the area of no visible signs of impurities on working surface (Spectrum 3 on Figure 4). Meanwhile area with slightly visible signs of impurities (Spectrum 1 on Figure 4) was characterized with insignificant changes of carbon ($p > 0.05$) and titanium ($p > 0.05$) amounts but amount of nickel decreased significantly ($p < 0.05$), and oxygen increased ($p < 0.05$). Elements content within area with pronounced visible signs of impurities (Spectrum 2 on Figure 4) characterized by statistically argued changes

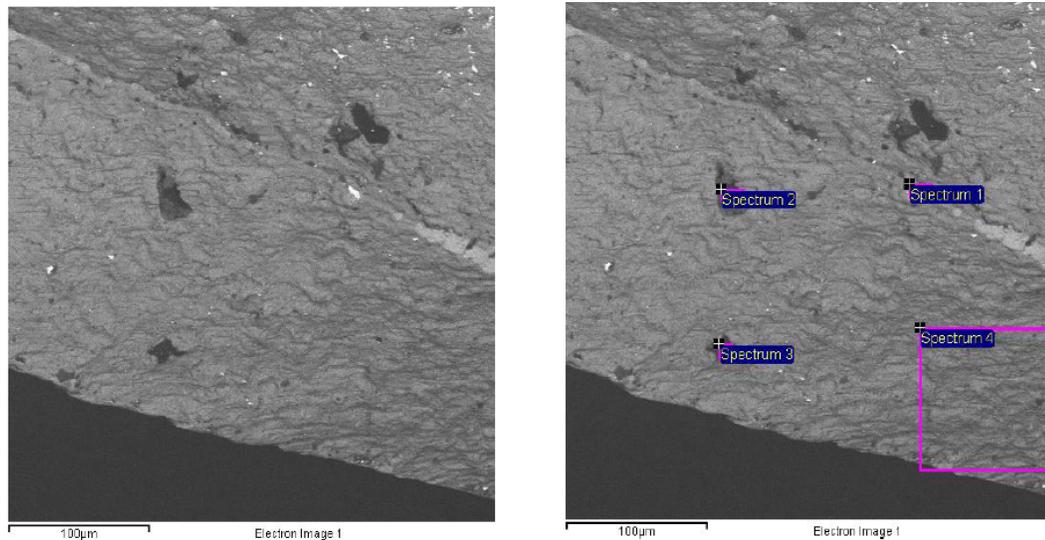


Figure 3: SEM image HyFlex CM file's working surface with markings of areas, which were analyzed for local element content through EDS (Spectrum 1 – area with no visible signs of impurities, Spectrum 2 – area with slightly visible signs of impurities, Spectrum 3 – area with pronounced visible signs of impurities).

Table 3: Local Element Content Registered Through EDS Among Areas with Different Visible Signs of Impurities Along Working Surface of HyFlex CM

| Spectrum | C (wt%) | O (wt%) | Na (wt%) | Mg (wt%) | Al (wt%) | Si (wt%) | S (wt%) | Cl (wt%) | K (wt%) | Ca (wt%) | Ti (wt%) | Ni (wt%) | Au (wt%) | Total (wt%) |
|------------|---------|---------|----------|----------|----------|----------|---------|----------|---------|----------|----------|----------|----------|-------------|
| Spectrum 1 | 4.07 | 10.95 | | | 0.35 | 0.52 | | | | | 32.56 | 24.88 | 26.67 | 100.00 |
| Spectrum 2 | 36.03 | 27.99 | | | | 0.72 | 0.51 | | | | 25.94 | 8.80 | | 100.00 |
| Spectrum 3 | 44.82 | 29.39 | 2.02 | 1.13 | 0.21 | 1.80 | 0.60 | 1.39 | 1.59 | 0.33 | 11.97 | 4.76 | | 100.00 |

Note: Spectrum 1 – area with no visible signs of impurities, Spectrum 2 – area with slightly visible signs of impurities, Spectrum 3 – area with pronounced visual signs of impurities.

within carbon ($p < 0.05$), oxygen ($p < 0.05$), titanium ($p < 0.05$) and nickel ($p < 0.05$) amounts, and traces of other elements such as Na, Mg, Al, Si and S also could be found at this area.

EDS of Prerace file demonstrated that area with no visible signs of impurities characterized by the 66.04% of iron, 16.64% of chromium, 8.45% of nickel, and 6.39% of carbon (Spectrum 3 on Figure 5). Spectrum 2, which represented area with slightly visible signs of impurities, demonstrated significant decrease in chromium ($p < 0.05$), iron ($p < 0.05$) and nickel ($p < 0.05$), while also statistically approved increase of oxygen was registered ($p < 0.05$). Significant increase in carbon was noticed only in the area with visible pronounced signs of impurities (Spectrum 1) ($p < 0.05$), which also was associated with further decrease in chromium ($p < 0.05$), iron ($p < 0.05$) and nickel ($p > 0.05$). Nitrogen also has been discovered in significant amount in area with pronounced visible signs of impurities ($p < 0.05$), but sodium, silicon, chromium, chromium and sulfur only demonstrated small traces.

None of the analyzed files was free of impurities over their working surface. Expressiveness of visible signs of impurities present over working surfaces of the files demonstrated correlation with nickel ($r=-0.62$, $p < 0.05$) and titanium ($r=-0.49$, $p < 0.05$) content at analyzed areas, while also with carbon ($r=0.57$, $p < 0.05$) and oxygen ($r=0.50$, $p < 0.05$) content. Odds ratio for possibility to register traces of one of the following elements N, Na, Mg, Al, Si, S, Cl, Cr, Fe, Ca at the working surface of endodontic file under condition of present pronounced visible signs of impurities was found to be at the level of OR=12.5 (95% CI: 1.08; 143.44; $p=0.04$).

DISCUSSION

Provided SEM-EDS analysis has revealed that working surfaces of five new Ni-Ti endodontic files contains areas with various visible signs of impurities, but none of the analyzed file demonstrated totally clean surface. In present study SEM images were

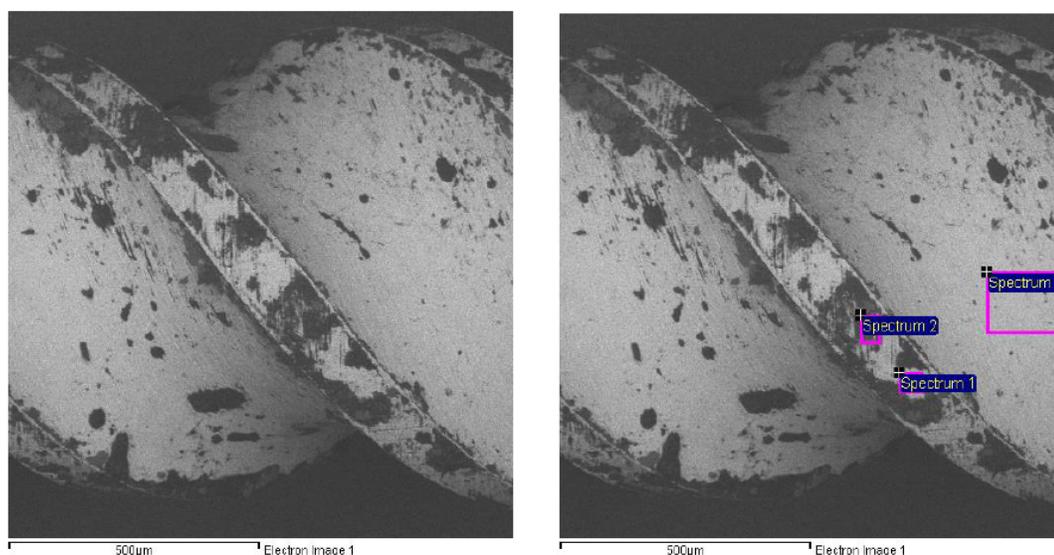


Figure 4: SEM image Profile file's working surface with markings of areas, which were analyzed for local element content through EDS (Spectrum 1 – area with slightly visible signs of impurities, Spectrum 2 – area with pronounced visible signs of impurities, Spectrum 3 – area with no visible signs of impurities).

Table 4: Local Element Content Registered Through EDS Among Areas with Different Visible Signs of Impurities Along Working Surface of Profile

| Spectrum | C (wt%) | O (wt%) | Na (wt%) | Mg (wt%) | Al (wt%) | Si (wt%) | S (wt%) | Ti (wt%) | Ni (wt%) | Total (wt%) |
|------------|---------|---------|----------|----------|----------|----------|---------|----------|----------|-------------|
| Spectrum 1 | 11.53 | 5.00 | | 0.56 | | 0.63 | 0.44 | 35.24 | 46.59 | 100.00 |
| Spectrum 2 | 54.69 | 23.99 | 0.65 | 1.56 | 0.23 | 2.14 | 0.57 | 10.29 | 5.89 | 100.00 |
| Spectrum 3 | 8.29 | | | | | | 0.36 | 37.93 | 53.42 | 100.00 |

Note: Spectrum 1 – area with slightly visible signs of impurities, Spectrum 2 – area with pronounced visible signs of impurities, Spectrum 3 – area with no visible signs of impurities.

conditionally categorized on areas with pronounced visible signs of impurities, areas with slightly visible signs of impurities, and areas with no visible signs of impurities. None of the above mentioned areas were characterized with the presence of solely nickel and titanium, even areas with no visible signs of impurities always demonstrated presence of some amount of carbon, oxygen, and potentially other elements in the minimal amount (except aurum in case of HyFlex CM file). It was noticed that visual expressiveness of impurities was associated with changes of nickel, titanium, oxygen and carbon content, while also with the possibility of other elements' traces identification.

Previous laboratorial analysis based on the SEM method revealed that surface of Ni-Ti rotary files are usually contains 62% of carbon, 30% of oxygen, and just 2% and 55% of nickel and titanium, respectively [13]. But authors also mentioned that elements content is changing from surface to the core of the instrument with increasing trend toward amount of nickel and titanium [13]. High amount of carbon and oxygen over

the Ni-Ti files surface may be argued by the contact with an air, while traces of other elements like silicon, magnesium and calcium may be interpreted as environmental contaminants sedimented during file's manufacturing process [13, 15].

Observational study has reported that endodontic files of different brands contains wide ranges of chemical elements along their working surface, but till now it was unclear how amount of such elements are related with visible signs of surface impurities registered via SEM analysis [6]. In present study specific levels of correlations were established between expressiveness of visual signs of impurities present over working surface and content of Ni, Ti, O and C. Also it was statistically proven that visible signs of surface impurities may be used to predict the presence of N, Na, Mg, Al, Si, S, Cl, Cr, Fe, Ca elements over working surface. In future it would be perspective to find out how presence of different chemical elements may impact clinical performance of Ni-Ti rotary instruments. On the other hand presence of surface

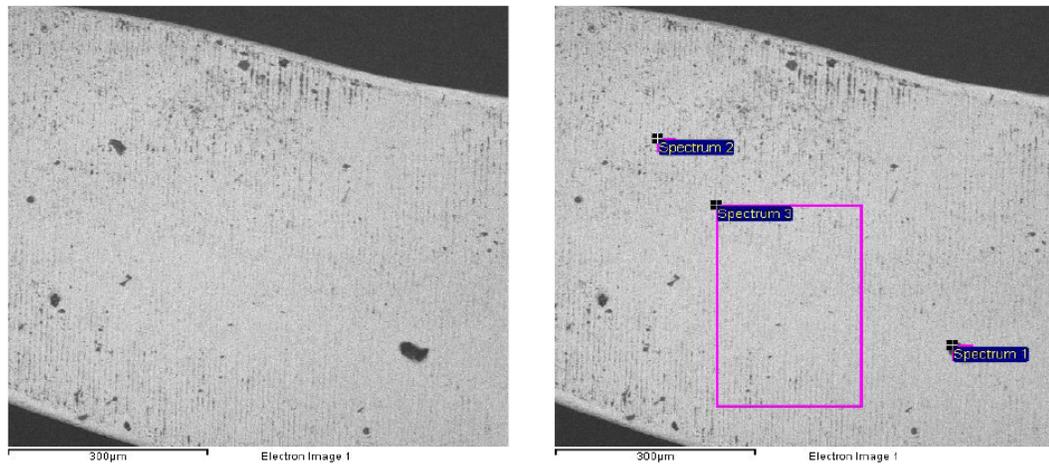


Figure 5: SEM image of Prerace file's working surface with markings of areas, which were analyzed for local element content through EDS (Spectrum 1 – area with pronounced visible signs of impurities, Spectrum 2 – area with slightly visible signs of impurities, Spectrum 3 – area no visible signs of impurities).

Table 5: Local Element Content Registered Through EDS Among Areas with Different Visible Signs of Impurities Along Working Surface of Profile

| Spectrum | C (wt%) | N (wt%) | O (wt%) | Na (wt%) | Mg (wt%) | Al (wt%) | Si (wt%) | S (wt%) | Cl (wt%) | Cr (wt%) | Fe (wt%) | Ni (wt%) | Total (wt%) |
|------------|---------|---------|---------|----------|----------|----------|----------|---------|----------|----------|----------|----------|-------------|
| Spectrum 1 | 48.25 | 12.53 | 19.99 | 0.24 | 0.82 | | 1.38 | 0.46 | 0.72 | 3.07 | 10.75 | 1.78 | 100.00 |
| Spectrum 2 | 8.23 | | 20.39 | | 10.24 | 3.59 | 7.95 | | | 10.48 | 36.67 | 2.45 | 100.00 |
| Spectrum 3 | 6.39 | | 1.90 | | | | 0.58 | | | 16.64 | 66.04 | 8.45 | 100.00 |

Note: Spectrum 1 – area with pronounced visible signs of impurities, Spectrum 2 – area with slightly visible signs of impurities, Spectrum 3 – area no visible signs of impurities.

impurities and traces of different elements may be used to categorize quality of Ni-Ti files manufacturing and packaging processes considering that all of above-mentioned elements may be categorized as environmental contaminants [6].

Medojevic *et al.* demonstrated that clean endodontic instruments represent solely presence of nickel and titanium (in ratio of 55.7:44.3%), while impurities on the files' working surfaces were associated with variety of elements [12]. In present study such elements as C, O and some other minor traces were found even at the areas of working surfaces, which did not present any visual signs of impurities. Such difference in obtained outcomes could be argued by the fact that different endodontic instruments were analyzed within two studies.

Another study of Medojevic *et al.* demonstrated that none of the analyzed specimen of endodontic manual file was free of surface impurities, containing different elements [14]. Such results are in correspondence with data obtained in our study, since SEM and EDS

revealed that every approbated file regardless of brand contained impurities over working surface, visible signs of which were various.

Elemental analysis provided by Hamdy *et al.* revealed that new Ni-Ti instruments characterized with predominant presence of titanium and nickel within its bulk structure, while authors acknowledged that levels of oxygen and carbon also could be found [15]. In present study areas of working surface with no visible signs of impurities also were represented mostly by nickel and titanium elements, while carbon and oxide were in minority, but situation was opposite within areas with visible signs of impurities present.

Kalyoncuoğlu *et al.* demonstrated that no significant Ni:Ti content changes have been observed after usage of rotary instruments compare to the situation before their use [18]. On the other hand authors did not report presence of any other elements over working surface of endodontic file [18], meanwhile in our study it was found that such even in places with no visible signs of impurities contains carbon, oxygen and minor traces of

other elements. Hypothetically amount of such may change during the use, even if correspondence between nickel and titanium remains the same.

Potentially areas of impurities accumulation may serve as retentive zones for debris adhesion during active file usage, which in turn may increase a risk of file separation during endodontic treatment [6]. Mechanism of such consequences are still not argued with sufficient evidences and requires more detailed and targeted researches. Some studies reported that amount of surface impurities may be associated with prevalence of surface defects presented in various forms over working part of endodontic files [6, 11, 13, 15]. Combination of both surface defects and surface impurities may affect file's condition in significant manner during its clinical usage and potentially compromise its functional prognosis over repetitive mode of action.

Limitation of present study associated with the fact that EDS and local elements content analysis were provided only over few randomly chosen areas of working surfaces, which by subjective grading of operator contains no visible signs of impurities, slightly visible signs of impurities and pronounced visible signs of impurities. In future it would be beneficial to provide analysis over all working surface of the file and to detail how local elements content differs over various file surfaces and localizations with the same and different visual signs of impurities accumulation. On the other hand advantage of this study is related with the fact that files and surfaces were chosen randomly, and obtained results potentially may represent general situation with impurities over files' working surfaces. Another limitation of the study is caused by analysis provided only over surface of endodontic instrument, meanwhile it would be beneficial to assess how element content is changing from the surface to the core of the file, and how it may potentially affect Ni-Ti file performance during its clinical usage.

CONCLUSION

Five different new Ni-Ti endodontic files demonstrated presence of various chemical elements over its working surface, but not just a presence of nickel and titanium. Pattern of chemical impurities accumulation and distribution over working surfaces of five new Ni-Ti files was diverse and characterized with different levels of chemical elements amounts among different files and among different areas with visually variously expressed signs of impurities over the working surface of the same file. Carbon, oxygen,

nickel, and titanium were the most prevalent elements observed along working surfaces of five new different endodontic files, while some instruments were characterized with the presence of tungsten, silicon, aurum, iron, chromium, magnesium and nitrogen, maximum level of which at selected areas with pronounced visible signs of impurities outreach 10% weight percent values. Areas with pronounced visible signs of impurities observed over SEM images of files' working surfaces were characterized also with greater changes of local elements content compared to areas with slightly visible signs of impurities, if areas with no visible signs of impurities were used as a reference.

DECLARATION OF INTEREST

The author reports no conflict of interest, and the article was not funded or supported by any research grant.

REFERENCES

- [1] Liang Y, Yue L. Evolution and development: engine-driven endodontic rotary nickel-titanium instruments. *Int J Oral Sci.* 2022; 14(1): 12. <https://doi.org/10.1038/s41368-021-00154-0>
- [2] 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>
- [3] Silva EJ, Martins JN, Lima CO, Vieira VT, Fernandes FM, De-Deus G, Versiani MA. Mechanical tests, metallurgical characterization, and shaping ability of nickel-titanium rotary instruments: a multimethod research. *J Endod.* 2020; 46(10): 1485-94. <https://doi.org/10.1016/j.joen.2020.07.016>
- [4] Grande NM, Castagnola R, Minciaccchi I, Marigo L, Plotino G. A review of the latest developments in rotary NiTi technology and root canal preparation. *Austr Dent J.* 2023; 68: S24-38. <https://doi.org/10.1111/adj.12998>
- [5] Gambarini G, Miccoli G, Zanza A, Del Giudice A, Testarelli L. How to Improve Properties of Nickel-Titanium Rotary Instruments. *J Comtemp Dent Pract.* 2020; 21(4): 351-2. <https://doi.org/10.5005/jp-journals-10024-2796>
- [6] Noenko I, Goncharuk-Khomyn M. Scanning Electronic Microscopy Surface Characteristics of Six Endodontic Files Systems Available in Ukraine: Observational Study. *J Int Dent Med Res.* 2023; 16(1): 24-31.
- [7] Noenko I, Goncharuk-Khomyn M, Belun V, Biley A. Counterfeit Endodontic Files Features Objectified with Scanning Electronic Microscopy: Comparative Study of SOCO SC Pro Original and Falsified Rotary Instruments. *J Int Dent Med Res.* 2023; 16(2): 565-573
- [8] Martins JN, Silva EJ, Marques D, Baruwa AO, Caramês J, Braz Fernandes FM, Versiani MA. Unveiling the Performance of Nickel-Titanium Endodontic Instruments through Multimethod Research: A Review. *Appl Sci.* 2023; 13(12): 7048. <https://doi.org/10.3390/app13127048>
- [9] Gomes MS, Vieira RM, Böttcher DE, Plotino G, Celeste RK, Rossi-Fedele G. Clinical fracture incidence of rotary and reciprocating NiTi files: A systematic review and meta-regression. *Aust Endod J.* 2021; 47(2): 372-85. <https://doi.org/10.1111/aej.12484>

- [10] Gavini G, Santos MD, Caldeira CL, Machado ME, Freire LG, Iglecias EF, Peters OA, Candeiro GT. 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>
- [11] AbuMostafa A, AlOmari M, AlQashtini I, AlAbdullah S, AlJabr N, Domia R. Perfection of unused Ni-Ti endodontic files, myth or reality? A scanning electron microscope (SEM) study. *J Dent Oral Hyg.* 2015; 7(2): 16-21. <https://doi.org/10.5897/JDOH2014.0138>
- [12] Medojević MJ, Zdravković A, Medojević M, Stratimirović Đ. Evaluation of surface characteristics of new rotary nickel-titanium instruments-SEM-EDS analysis. *Serb Dent J.* 2021; 68(1): 19-30. <https://doi.org/10.2298/SGS2101019J>
- [13] Bonaccorso A, Tripi TR, Cantatore G, Condorelli GG. Surface properties of nickel-titanium rotary instruments. *Endod Prac Today.* 2007; 1(1): 45-52.
- [14] Jovanović-Medojević M, Pelemiš M, Nešković J, Popović-Bajić M, Stratimirović Đ, Živković S. Analysis of working surface in new manual and rotary endodontic instruments (scanning electron microscopy). *Srp Arh Celok Lek.* 2020; 148(7-8): 398-403. <https://doi.org/10.2298/SARH190704018J>
- [15] Hamdy TM, Galal M, Ismail AG, Abdelraouf RM. Evaluation of flexibility, microstructure and elemental analysis of some contemporary nickel-titanium rotary instruments. *Open Access Maced J Med Sci.* 2019; 7(21): 3647. <https://doi.org/10.3889/oamjms.2019.811>
- [16] Laboratory of electron microscopy – NanoMEDTech. <https://nanomedtech.ua>. (accessed February 1, 2024).
- [17] Hannigan A, Lynch CD. Statistical methodology in oral and dental research: pitfalls and recommendations. *J Dent.* 2013; 41(5): 385-392. <https://doi.org/10.1016/j.jdent.2013.02.01>
- [18] Kalyoncuoğlu E, Keskin C, Uzun İ, Bengü AS, Guler B. Scanning electron microscopy with energy dispersive X-ray spectrophotometry analysis of reciprocating and continuous rotary nickel-titanium instruments following root canal retreatment. *J Oral Sci.* 2016; 58(3): 401-6. <https://doi.org/10.2334/josnurd.15-0725>

Received on 02-01-2024

Accepted on 16-02-2024

Published on 20-02-2024

DOI: <https://doi.org/10.12974/2311-8695.2024.12.01>

© 2024 Noenko *et al.*

This is an open access article licensed under the terms of the Creative Commons Attribution Non-Commercial License (<http://creativecommons.org/licenses/by-nc/3.0/>) which permits unrestricted, non-commercial use, distribution and reproduction in any medium, provided the work is properly cited.