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SURFACE TOPOGRAPHY AND CHEMICAL CHARACTERISTICS OF ROTARY NI TI FILES OF DIFFERENT MANUFACTURING TECHNIQUES AFTER MULTIPLE USE

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ABSTRACT

Background: This study designed to evaluate and compare surface physical and chemical characteristics of recently introduced rotary NiTi files Neolix (Neolix, Châtres-la-Forêt, France), One Curve (Micro mega, France) and AF™ Blue S ONE File System (Fanta, USA) before and after 1st and 3rd use.

Materials and Methods: In this study thirty brand-new endodontic files having tip size 25/0.6 of the following systems were used: Neolix (Neolix, Châtres-la-Forêt, France), One Curve (Micro mega, France) and AF™ Blue S ONE One-File System (Fanta, USA). These files were equally divided into three groups (n=10). Ninety simulated curved canals (Endo Training Block-L; Dentsply Maillefer, Ballaigues, Switzerland) with curvature of 45º±10º, with 0.02taper, 0.15 mm apical diameter, and 16 mm length were used. File`s surfaces examined by Environmental Scanning Electron Microscope ESEM (FEI Quanta 250 FEG, Berlin, Germany) at 500X magnification before use. Cutting blade (active part) images been processed and their micrographs were analyzed at the tip, 2 and 4 mm short of its cutting tip. The Nickel (Ni) and Titanium (Ti) average contents (wt %) on the instrument`s surface were also analyzed before their use, using an Energy Dispersive Spectrometer Device (EDAX - AMETEK, Inc., Mahwah, NJ, USA). All instruments prepared using electric motor (Motor X-Smart Plus, Dentsply/Maillefer), following manufacturer`s instructions for each system. Each used file prepared three simulated canals, after each canal instrumentation, each file was removed for cleaning its flutes gently with sterile gauze, and for canal irrigation using with 2 mL/ 2.5% NaOCl irrigating solution. After the first and third instrumentation performed; files were cleaned, dried, stored and sent to SEM equipment for post-instrumentation files surface's topography analysis and chemical composition of the NiTi content (wt %) according to the previously described pre-instrumentation analysis protocol. SEM micrographs recorded on a CD-ROM and projected for three blinded examiners'. They were given a spreadsheet including the instrument data (number, the location to be evaluated; tip, 0-2mm or 2-4mm from the tip) and the side of the instrument (detail facing up or down), and the criteria of defects and deformation assessment namely (irregular edges, grooves, micro-cavities and burrs).Data were recorded and statistically analyzed.

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Results: Analysis of surface defects (SEM) showed that; all file systems have pre-instrumentation surface defects which increased after use. Neolix file had highest significant difference in all detected surface defects with One Curve file, with AF™ Blue S ONE file in burs and micro-cavities at file`s Tip and 0-2mm, in all examination intervals($p<0.05$). One Curve file showed the least surface defects with significant difference with AF™ Blue S ONE file in edges mean values at file's Tip at all examination intervals. EDS chemical analysis of Ni and Ti% revealed; decrease in Ni, Ti weight % after first and third uses. Decrease in Ni% was more than Ti%.

Conclusion: surface defects of the studied instruments were recorded before use, continuous use promoted their increase. Chemical change results revealed significant changes in Ni and Ti content on instruments surface after reuse led to that may affect their performance.

KEY WORDS: surface topography, chemical characteristics, SEM, EDAX, Neolix, One Curve and AF™ Blue S ONE

INTRODUCTION

The innovative Nickel-titanium alloy (NiTi) was developed in 1960's owns amazing mechanical properties "superelasticity and shape memory", their flexibility can be inferred to its lower modulus of elasticity compared to stainless steel, while the ductility of the alloy is responsible for its superior fracture resistance [(1)]. In 1988 ***Walia et al.*** put in the first manual NiTi files in endodontic field [(1)]. The increased popularity of NiTi rotary files have manifested in its safe, efficient, and well adaptation to shape root canals with severe curvature [(2,3)]. As the popularity of traditional NiTi files increased, manufacturers have focused their efforts on the improvement of NiTi performance to make them efficiently used, resist deformation and fracture [(3)], to overcome their unexpected failures happened regardless of visibly permanent plastic deformation free surface [(4)]. Various approaches of files cross sectional design modification proved have ability to alter the mechanical features of rotary files [(5)]. One the other hand; manufacturing of these NiTi rotary files requires a special procedure, and could result in an increased surface defects concentration including; debris, pits, metal strips and blunt cutting edges [(6)]. Therefore, different manufacturing strategies including; heat treatment processes and different thermo-mechanical treatments, have been continuously intended for Ni-Ti instruments mechanical properties improvement, as; fatigue fracture resistance, flexibility, and risk of fracture [(7,8)]. New single-file systems have innovated in endodontic practice utilizing either rotation or reciprocation motions gained huge popularity as they include: only a single file that require a minimum or no glide path to complete instrumentation of root canals, the recommendation for use the file only once has gained advantage of minimizing instrument fatigue and eliminating cross contamination between patients, and reducing the working time [(9, 10)]. Newly introduced full rotary motion single-file systems investigated in this study include; Neolix (Neolix, Châtres-la-Forêt, France) that made up of special alloy with a non-homogeneous rectangular cross section that enhances the file flexibility. According to the manufacturer it offers many advantages such as sharp cutting edges, Gothic-like tip design and built-in abrasive properties [(11)]. It is manufacturing utilizing a recently introduced wire-cut electrical discharge machining (WEDM) mechanism, which creates a rough surface, adding abrasive properties that increase root canal preparation speed. These files undergo suitable heat treatment which earn the instrument higher flexibility and shape memory [(12)]. Another recently introduced single rotary file is the One Curve (Micro mega, France) file that is manufactured from heat-treated Nickel-Titanium alloy (C. Wire). The manufacturer claims, this file has a unique manufacturing technique that implemented by MICRO-MEGA for developing a controlled memory NiTi files that can be pre-bend for easier instrumentation of the root canal and elimination of difficulties, increased blade flexibility and more fracture resistance for higher overall security. Moreover, it owns cross-section varies along the blade for improving the centering ability in the root canal apical third and removal of debris reaching middle and coronal parts [13]. A more recently, AF™ Blue S ONE File System (Fanta, USA) was introduced; it is manufactured from specially heat treated wire AF wire (AF™-H). According to the manufacturer, it has excellent mechanical strength properties, greater resistance to cyclic fatigue, and its flexibility is enough to avoid canal transportation. Meanwhile, its hardness is large enough to allow for good cutting efficacy. Flawless surface finishing, minimum radial contact to ensure better cutting ,variable cross-section on file, increase the volume for upward debris eliminations, together with the variable pitch enables efficient debris transport and reduces the screwing effect[14]. Up to our knowledge**,** there are no studies investigated the surface physical and chemical changes that occur on studied instruments surface after their repeated use. Therefore, this study designed to evaluate surface characteristics and chemical composition changes of NiTi alloy of these instruments before and after three times use.

MATERIALS AND METHODS

Thirty brand-new endodontic files have tip size 25/0.6 of the following systems were used in this study: Neolix (Neolix, Châtres-la-Forêt, France), One Curve (Micro mega, France) and AF™ Blue S ONE One-File System (Fanta, USA). Ninety simulated canals (Endo Training Block-L; Dentsply Maillefer, Ballaigues, Switzerland) with curvature of 45º±10º, a taper of 0.02, an apical diameter of 0.15 mm, and length of 16 mm were used in this study. Files were equally divided into three groups (n=10) according to type of the file system as follow; Group 1- Neolix, Group 2- One Curve and Group 3- AF™ Blue S ONE. In order not to interfere in the results of the experiment; files were handled from their packaging carefully using clinical tweezers, were held by their shank; no pre-use instruments cleaning was performed, as they were sterile.

Pre-instrumentation files surface's analysis

Files were fixed at the shank using utility wax on the SEM holder for been initially analyzed of their topography by Environmental Scanning Electron Microscope ESEM (FEI Quanta 250 FEG, Berlin, Germany) at 500X magnification. using one point in instrument shank (groove detail facing up) as a reference for image reading of the cutting blade (active part) of each file, and the second set of images with the groove detail facing downwards so that the active portion of the file could be seen on both sides. After visualization of each file, the images of the cutting blade (active part) were processed and analysis of the instruments active part micrographs including; the tip, 2 mm and 4 mm short of the cutting tip, at 500X magnification. The Ni& Ti average (wt %) content on the instruments surface before their use was also analyzed, by using an energy dispersive spectrometer (EDS) (EDAX - AMETEK, Inc., Mahwah, NJ, USA). One EDX spectrum was collected at the tip of each file, as it represents most deformations after use [15] under the following situation: 25 kV accelerating voltage, 110 μ A beam current, 10^{-6} Torr pressure (high-vacuum), analysis area of 130×130 μm at magnification 1000, acquisition time 200 s and detector dead time 30–35%. Nonstandard analysis mode used to perform the quantitative analysis, utilizing ZAF correction methods, by Genesis software (version 5.2; EDAX) [16]. After SEM micrographs taking and EDS readings, the instruments were stored in polypropylene closed numbered tubes.

Simulated Root Canal Preparation

Working length of the simulated canals was determined using a K-type file size 10 (Dentsply/ Maillefer) that inserted into the simulated canal, until visualized its tip at the canal end. Then, it was withdrawn to 1 mm shorter than obtained length, determining 15 mm working length. investigated instruments were mounted in a contra-angle has 6:1 gear reduction (Dentsply/Maillefer), powered by an electric motor (Motor X-Smart Plus, Dentsply/ Maillefer), at the setting previously identified for each used system as follow; Group 1- simulated canals were prepared using Neolix files powered with speed of 300 rpm and torque of 1.5 N/ cm, according to manufacturer's instructions in circumferential brushing action until reached the working length. In Group 2 simulated canals were prepared using One Curve files at speed of 300 rpm, 2.5 N/cm torque according to manufacturer's instructions using direct downward movement in three waves till reaching the working length. Group 3 simulated canals were prepared using AF™ Blue S ONE files powered by the electric torque control motor at speed 350 rpm, Torque 2.6 N/cm using a gentle inward motion down to the working length. Single operator, a specialist in endodontics prepared all root canals in all groups. After each three pecks, the files were removed for cleaning its flutes gently with sterile gauze, and for canal irrigation using 2 mL /2.5% NaOCl solution utilizing a 27-gauge irrigation needle (Dentsply Maillefer, Shanghai, China). Each file was used in three canals preparation, after each canal instrumentation, the files were submitted to thermo-chemical cleaning in an ultrasonic bath (Cristófoli, Campo Mourão, PR, Brazil) using a heating system/ 10 min at 65 ºC with water/enzymatic detergent Endozime (DFL) at a ratio of 5 mL/liter of distilled water.

Post-instrumentation files surface's analysis

Following first and third instrumentation; the instrument properly dried and stored in polypropylene numbered, closed tubes and sent to SEM equipment for post-instrumentation files surface's topography analysis and NiTi content (wt %) chemical analysis using EDS on the instruments surface according to the previously described pre-instrumentation analysis protocol.

Analysis of SEM micrographs

All ESEM micrographs of different intervals (pre-instrumentation, after first use, and after third use) were saved on a CD-ROM and applied into PowerPoint program, numbered, and then were projected for three blinded specialized examiners' observation. These examiners were previously calibrated (0.88 and 0.90 - Kappa test). They delivered a spreadsheet includes the number of the examined instrument, the level of evaluation (tip, 0-2mm or 2-4mm) and the instrument side to be examined (detail facing up or down), and the criteria for evaluation of each defects and deformation types namely (irregular edges, grooves, micro-cavities and burrs). Using ***Troian et al***.[17] scoring system that was adopted for topographic changes of the instruments classification as follow: 1 - the instrument long axis examined surface has no defect or deformation; 2 - the instrument long axis examined surface with approximately one to three areas with defects or deformations; 3 - the instrument long axis examined surface with approximately four to five areas with defects or deformations; 4 - the instrument long axis examined surface with over five areas with defects or deformations. Examiners were formerly instructed that if they had any doubt between scores, the higher score should be chosen. ***Statistical Analysis;*** obtained data submitted to the normality test (Kolmogorov & Smirnov test) for analyzing the differences in the mean chemical content of the Ni and Ti (wt %) in the instrument surface and to analyze the differences in mean surface defect values between groups and subgroups, the obtained values were statistically analyzed using 1-way ANOVA and Tukey post-hock test ($p<0.05$). While, the Kruskal- Wallis test ($p<0.05$) was performed to analyze the differences between the mean scores of total surface topographic changes in the instruments surfaces. SPSS program for windows (version.20) used to perform statistical tests.

RESULTS

Analysis of Instruments Surface`s Defects (SEM)

Statistical analysis of recorded surface defects in all examined instruments (***Neolix , One Curve and AF™ Blue S ONE***) revealed occurrence of defects (**IE**: Irregular Edges, **B**: Burrs, **LG**: Longitudinal Grooves and **MC**: Micro-Cavities) before and after reuse. Regarding each instrument there was non-significant difference of each examined defect at each examined level individually (Tip, 0-2mm, and 2-4mm)in all examination interval (before,1st and, 3rd use). The incidence of defects and deformations observed on the instruments surface before and after the first and third use is in Table 1. Representative SEM micrographs of the examined levels of the instruments (Tip, 0-2mm, and 2-4mm) are in Figure 1.

Statistical analysis of differences between the mean defects values of the examined levels (tip, 0-2 and 2-4) in each file (NEO, OC, AF) individually at each examination interval showed that; in the ***Neolix files*****; MC *mean values*** were the highest among all the recorded defects. Significant differences were recorded between Tip and 0-2mm levels before use also, between tip and 2-4mm after 1st use. The 2-4mm level was significantly lower than 0-2 and tip levels after 3rd use examination interval. On the other hand, the **LG** mean values showed non-significant difference between all examination levels at different examination intervals. **The IE** mean values showed that the 2-4 level was significantly lower than the Tip and 0-2 levels at 1st and 3rd examination interval. While, the **B** recorded lowest defect mean values, with significant difference between all examination levels before and after 1st use examination intervals only. ***The One Curve***

TABLE (1) Comparison between study groups according to Mean±SD of defects up to 4mm of their active part; before, after 1st and 3rd use.

Defect	File level	G.1: Neolix (n=10)			G.2: One Curve(n=10)			G.3: AF-Blue (n=10)			p value
		Before	1st	3rd	Before	1st	3rd	Before	1st	3rd	
IE	Tip	$5.33^{A}\pm0.58$	$5.67^{AX}\pm0.58$	$5.67^{AX}\pm0.58$	0 ± 0^{B}	0 ± 0^{B}	0^{B}	$3.67^{A}\pm0.58$	$3.67^{AX}\pm0.58$	$3.67^{AX}\pm0.58$	≤0.05
	0-2	$3.33^{A}\pm0.58$	$4.33^{AX}\pm0.58$	$4.33^{AX}\pm0.58$	0 ± 0^{B}	$0.33^{B}\pm0.58$	$00.33^{B}\pm0.58$	$1.33^{B}\pm1.53$	$1.33^{BY}\pm1.53$	$1.67^{BXY}\pm2.08$	≤0.05
	2-4	$1.33^{A}\pm0.58$	$1.67^{AY}\pm1.15$	$2.33^{AY}\pm0.58$	0 ± 0^{A}	0 ± 0^{A}	0 ± 0^{B}	0 ± 0^{A}	0 ± 0^{AY}	0 ± 0^{BY}	≤0.05
p- value		NS	≤0.05	≤0.05	NS	NS	NS	NS	≤0.05	≤0.05	
B	Tip	$4.67^{AX}\pm0.58$	$5.00^{XA}\pm0.00$	$5.00^{A}\pm0.00$	0 ± 0^{B}	0 ± 0^{B}	0 ± 0^{B}	$0.68^{B}\pm0.58$	$0.67^{B}\pm0.58$	$0.67^{B}\pm0.58$	≤0.05
	0-2	$2.33^{AY}\pm0.58$	$2.67^{YA}\pm0.58$	$3.00^{A}\pm0.00$	0 ± 0^{B}	$0.33^{B}\pm0.58$	$0.33^{B}\pm0.58$	0 ± 0^{B}	0 ± 0^{B}	0 ± 0^{B}	≤0.05
	2-4	$0.33^{AZ}\pm0.58$	$0.67^{ZA}\pm0.58$	$0.67^{A}\pm0.58$	0 ± 0^{A}	0 ± 0^{A}	0 ± 0^{A}	0 ± 0^{A}	0 ± 0^{A}	0 ± 0^{A}	NS
p- value		≤0.05	≤0.05	NS	NS	NS	NS	NS	NS	NS	
LG	Tip	$7.00^{A}\pm0.00$	$8.00^{A}\pm1.00$	$8.67^{A}\pm1.53$	$4.00^{BC}\pm1.00$	$4.667^{BC}\pm1.16$	$5.00^{BC}\pm1.00$	$6.00^{AC X}\pm1.00$	$6.67^{ACX}\pm0.58$	$7.00^{AC}\pm1.00$	≤0.05
	0-2	$6.67^{A}\pm0.58$	$6.67^{A}\pm1.16$	$7.00^{A}\pm0.00$	$2.67^{B}\pm0.58$	$3.00^{BC}\pm0.00$	$3.00^{B}\pm0.00$	$5.00^{AX}\pm1.00$	$5.67^{ACXZ}\pm1.16$	$6.00^{A}\pm1.00$	≤0.05
	2-4	$6.33^{A}\pm0.58$	$6.67^{A}\pm0.58$	$7.00^{A}\pm0.00$	$2.00^{B}\pm1.00$	$2.667^{B}\pm1.53$	$2.67^{B}\pm1.53$	$3.00^{BY}\pm1.00$	$3.33^{BYZ}\pm0.58$	$3.33^{B}\pm0.58$	≤0.05
p- value		NS	NS	NS	NS	NS	NS	≤0.05	≤0.05	NS	
MC	Tip	$11.00^{XA}\pm0.0$	$13.33^{XA}\pm1.53$	$13.67^{XA}\pm1.16$	$8.00^{BCX}\pm1.0$	$8.33^{BC}\pm0.58$	$9.00^{B}\pm1.0$	$9.67^{AC}\pm0.58$	$10.33^{AC}\pm0.58$	$10.66^{B}\pm0.58$	≤0.05
	0-2	$8.67^{YZA}\pm0.58$	$10.3^{XZA}\pm0.58$	$10.7^{XZA}\pm1.53$	$5.33^{BY}\pm1.16$	$6.33^{B}\pm1.16$	$6.33^{B}\pm1.16$	$9.67^{A}\pm0.58$	$11.00^{A}\pm.1.00$	$11.33^{A}\pm0.58$	≤0.05
	2-4	$9.00^{XZA}\pm1.00$	$10.0^{YZA}\pm1.00$	$10.00^{YA}\pm1.00$	$5.00^{BY}\pm1.53$	$5.333^{B}\pm1.53$	$6.33^{BC}\pm1.53$	$7.67^{A}\pm0.58$	$8.667^{A}\pm1.16$	$8.67^{AC}\pm1.16$	≤0.05
p- value		≤0.05	≤0.05	≤0.05	≤0.05	NS	NS	NS	NS	NS	
p-value		0.001									

Mean values have same superscript capital letters (X,Y and Z) means non-significant difference between compared items within the same column.

***file*s analysis showed that; **MC** mean defect values were the highest among recorded defects, with significant difference only between Tip and 0-2, Tip and 2-4 examined levels before use. On the other hand, other recorded defects (**LG, IE,** and **B**) mean values showed non-significant.

***Regarding the AF™ Blue S ONE file*s; the MC, B** defects recorded the highest and lowest mean values respectively among recorded defects, **with** non-significant difference between all examined levels at all examination intervals. While the **IE** mean defect values showed significant difference only between Tip/0-2 and Tip /2-4 examination levels after 1st use examination interval, and between tip/ 2-4 at 3rd use interval. Also, the **LG** mean defect values at 2-4 level were significantly lower than the tip at 1st use interval.

The comparison between the tested files (Inter-groups) at each level and interval (use) revealed that;

***Neolix file*s and *One Curve file*s showed the**

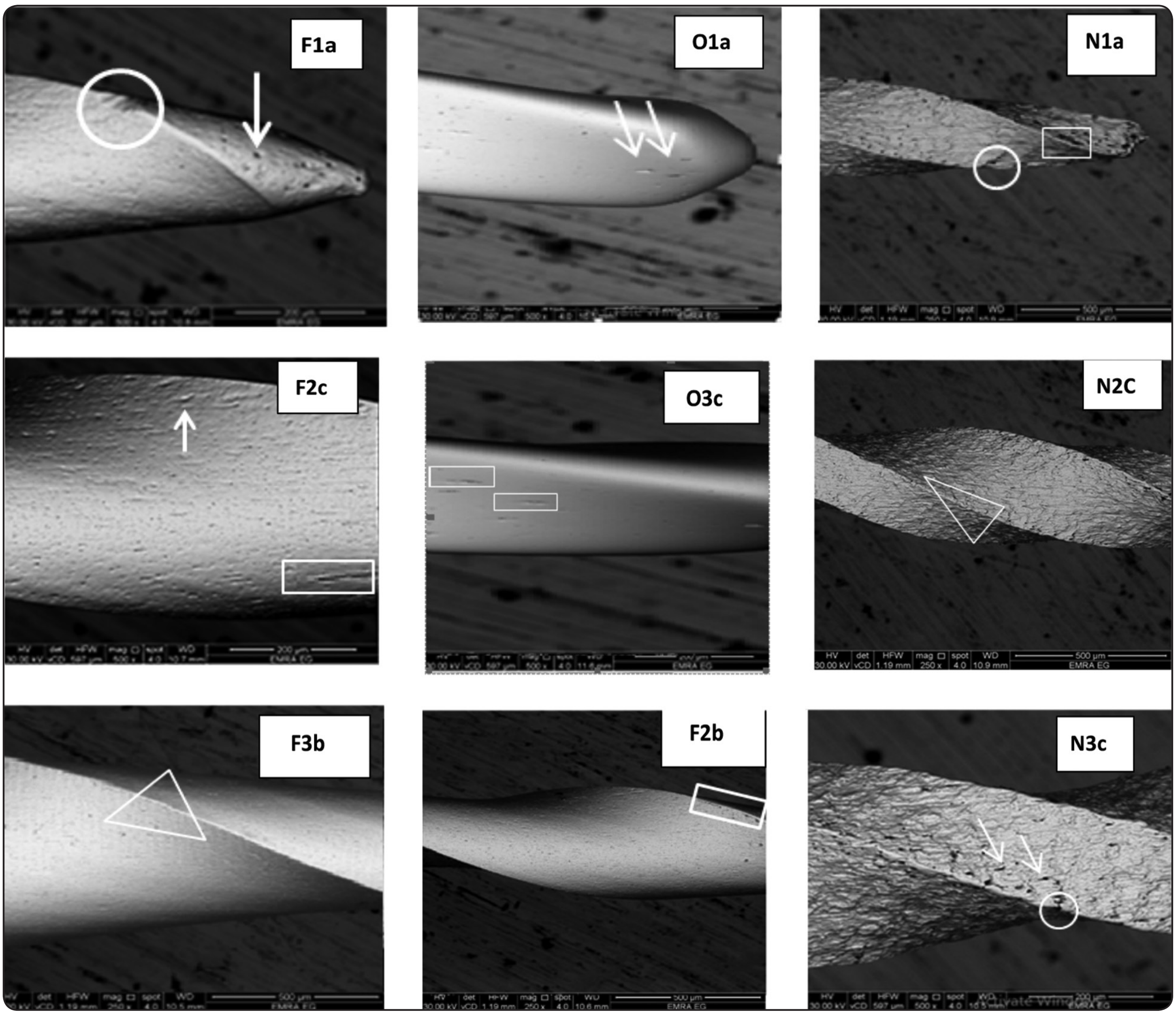

Fig. (1) Representative SEM micrographs of the instruments surface before, after 1st and 2nd uses. Magnification ×500, (arrow) Micro-cavities, (circle) Irregular Edges, (triangle) Burrs, and (box) Longitudinal Grooves. Numbers denotes examination intervals (1; before,2; 1st and3; 3rd uses). Capital letters (F; AF file, O; One Curve and N; Neolix file). Small letters denotes examination levels (a; Tip, b; 0-2mm and c; 2-4mm).

highest and lowest mean surface defect values respectively at all level and intervals. ***The Neolix files*** recorded significant higher mean surface defect values of **LG, MC ,IE,** and **B** than ***One Curve files*** at all levels and intervals except at 2-4 level that showed no significant differences at before and 1st use for **IE** defect and at before,1st and 3rd use for **B** defect . Moreover, ***Neolix files*** showed higher significant mean defect values than ***AF™ Blue S ONE files*** regarding **IE** (in 0-2 level at all intervals, 2-4 level at 3rd use), **B** (in Tip and 0-2 level at all intervals), and **LG** (in 2-4 level at all intervals). ***On the other hand, the One Curve files*** showed significantly lower mean surface defect values than ***AF™ Blue S ONE file*** regarding the **IE** at file`s Tip at all examination intervals, **MC** at 0-2mm, in all examination intervals, and **LG** at 0-2 mm before and after 3rd uses.

The analysis of median score values for total surface defects revealed that; No significant differences were found between different intervals of use (pre,1st and 3rd uses) and levels within each tested file. Neolix files showed the highest statistically significant median score values at all levels and intervals.

Quantitative Analysis of Ni and Ti (EDS): The mean values (wt %) of Ni and Ti content on the instruments surface are in Figures 2. All examined file systems Neolix file, One Curve and AF™ Blue S ONE file had significant decrease in Ni content between all examination intervals (before use, after 1st and 3rd uses) $p<0.05$. Regarding Ti content, Neolix file and One Curve files the instruments had no significant decrease throughout use ($p>0.05$). However, AF™ Blue S ONE file, showed a significant decrease of Ti content between 3rd use interval with before and after 1st use values ($p>0.05$).

Inter-groups correlation revealed; non-significant difference in Ni % values between all files at examination intervals were noticed except, significant difference between One Curve and AF™ Blue S ONE files after 3rd used. No significant difference in Ti% values between Neolix file, One Curve and AF™ Blue S ONE file before use. However, AF™ Blue S ONE files showed significant lower Ti % than Neolix and One curve files at 1st and 3rd uses.

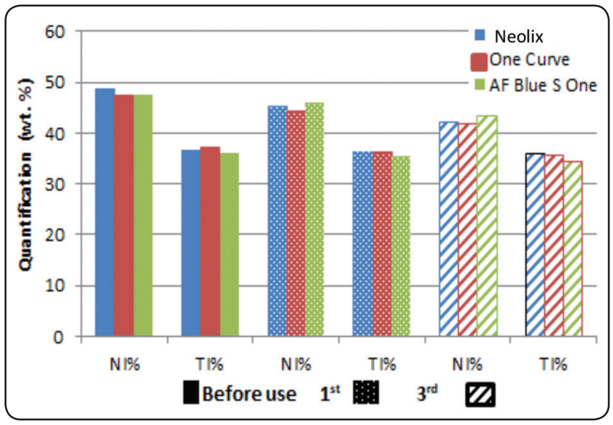

Fig. 2: Mean values of Ni and Ti quantification before and after 1st and 3rd instruments use.

DISCUSSION

Natural dentition retained functioning and esthetically accepted is the basic goal of endodontic treatment. Accomplishment of such goal relies on variety of steps starting from proper selection of the case and treatment plan up-to final restoration. ***Schilder*** [18] established clinical objectives of cleaning and shaping including development of a continuously tapered canal with maintaining its original shape and curvature. Instruments made from nickel-titanium alloy development considered as the leap in achieving such objectives owing to their super-elasticity and shape memory [1]. Despite of this; their sudden fracture is a clinical concern ``nightmare`` that has varied incidence from 3% to 21% [4, 19]. Therefore, awareness about the leading factors to instrument fracture is important which occurs even there is no visibly detected sign of defects, it shouldn't be ignored that operator`s proficiency as well as root canal morphology were recorded to have influential role on the fracture incidence [20,21]. On the other hand; improvement of

Ni Ti instrument`s flexibility and fatigue fracture became main concern for manufacturers through including different manufacturing process, thermo-mechanical treatments of the alloy and modifications in cross-sectional design [1, 22,23]. So, manufacturers switch to single use Ni-Ti rotary files as a favorable step for instrument failure prevention due to repeated use [24-26]. But, even brand-new instruments are not fortified against failure[27]. Instruments included in this study are recommended for single use according to their manufacturers. However, in our work we use them for three times per each file. As the cumulative influence of multiple clinical uses on instrument`s surface physical and chemical characteristics provides direct hint about their fatigue failure behavior and the unexpected fracture risk too [3, 28]. For group's standardization, tested files had same tip size and taper 25/6 %. Also, simulated used canals had the same curvature of 45º±10º, 0.02taper, 0.15 apical diameter mm and 16 mm length. Unfortunately, resin blocks don't mimic the clinical situation due to circular cross section that influenced torsional loads on the instruments [29]. On contrary; ***Schafer &Florek*** [30] recorded higher values of torsional loads for resin (simulated canals) during instrumentation than of dentine (natural canals), which might mean that the failure resistance in natural canals is probably even greater than produced in the resin of the simulated canal. In this study, file`s surface topography analysis performed using SEM which proved as an accurate tool for evaluating the morphological changes of metallic materials, as endodontic instruments repeatedly, without affecting their physical properties [31,32]. Rotary NiTi instruments evaluated in this research showed variable surfaces defects although they were brand new, which could be inferred to the process of instruments manufacturing. This is in accordance with ***Chianello et al.*** [33] who proved no topographic irregularities free NiTi instrument, and at least 2 to 7 defects were recorded on the surface even before use. In this study pre-instrumentation images showed; Neoniti file had significantly higher percentage of surface defects than other tested file systems (One Curve and AF™ Blue S ONE) which may be attributed to its manufacturing technique utilizing a recently developed process; wire-cut electrical discharge machining (WEDM) that gives a rough surface, leads to abrasive properties that aim to enhance the speed of root canal preparation[11,12]. On the other hand; One Curve file group showed the least pre-instrumentation defects which may inferred to its manufacturing technique, as manufacturer's claims it's made of heat treated Ni Ti wire (C. Wire) with a technique designed, developed and applied exclusively by MICRO-MEGA. The results showed that surface defects increased after 1st use and after 3rd use in accordance with other studies that confirmed surface defects existence on endodontic file before use, may procure more surface damage after use [8, 34]. Neoniti file group recorded the highest increase in surface defects after use; this could be related to its non-homogeneous rectangular cross section. While the One curve file has variable cross-section triple-helical to S shape and AF Blue S ONE has variable cross-section on file triangular to S shape. These findings were explained in previous studies stating that; instruments with rectangular or square cross-sectional design has less flexibility, less stress distribution of along its length and highest stress concentrations in comparison with a triangular and S-shaped cross-section being more vulnerable to plastic deformations [35.36]. On the other hand, it couldn't be ignored that non homogenous surface of NiTi instruments and their softer cutting edges than the core of instruments lead to a lower cutting efficiency, a higher wear and a higher frequency of exchange of files [37]. In this study; all tested groups recorded the highest pre-instrumentation manufacturing defect at file`s tip which came against the manufacturers claims that the tips of rotary instruments should be conic, smooth. This in accordance with ***Sattapan et al*** [15] who stated that as the file taper increased, more bulk is given than its tip which accounts for files fracture close to the tip clinically due to less strength. The surface of NiTi alloy files proved to be formed of titanium oxide (TiO2), less nickel oxide (NiO) quantities and metallic nickel (Ni2O3) [38] , this layer has an important role in preventing wear and corrosion of the alloy internal layer [38,22]. This unique chemical composition and geometry formed via a strong binary intermetallic bond (20 to 200 Kcal/ mol) and bonding force (equiatomic) between Ni &Ti atoms of NiTi alloys, proved to be changed by; thermodynamic process, submission to stresses during root canal preparation [27]. On the other hand; ***Kalyoncuo.lu Despite et al.*** [39] reported non-significant change of Ni & Ti content before and after instruments use which against results of this study where both Ni and Ti atoms recorded decrease after use, but the decrease in Ni content was significantly higher than Ti that comes in accordance with ***Otsuka & Ren*** [38] . In this study; Micro-cavities were the highest recorded defect in all examined file systems in all evaluation intervals which might be attributed to loss of Ni and Ti atoms showed by EDS analysis on instruments surface after reuse, that comes in accordance with; ***Bastos et al***[40], ***Otsuka & Ren*** [38] Who proved that by repeated instrument use deteriorates the surface oxide layer, compromise the NiTi alloy to corrosion, that deteriorates the intermetallic bond, and allow easier Ni electron loss by oxidation than Ti electrons. None of the investigated instruments presented fracture that might be attributed to performance of preparation by single operator ``endodontics specialist`` that seems to influence the failure resistance of the files ***Generali et al*** [41]. On the other hand; this may be related to advances in manufacturing techniques as follow; Neoniti subjected to proper heat treatment that leads to increase in flexibility and shape memory of this system, One Curve made of heat-treated Nickel-Titanium alloy (C. Wire) which is exclusively developed and implemented for controlled memory of NiTi and capacity to pre-bend, and AF™ Blue S ONE, is manufactured from specially heat treated wire AF wire (AF™-H), that has excellent mechanical strength properties, greater resistance to cyclic fatigue, and good cutting efficacy[14]. These results denote that rotary endodontic files manufacturing and packaging process are far from targeted. Therefore, manufacturers should develop refinements in the machining techniques, to diminish the surface defects and thereby increase the clinical performance.

CONCLUSION

Within the limitations of this study, no brand new instrument were free of surface defects, these defects increase after instruments put in use and after multiple use. Ni & Ti contents of instruments surface decreased after instruments use which negatively affects prevalence of surface defects.

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