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Evaluation of Flexibility, Microstructure and Elemental Analysis of Some Contemporary Nickel-Titanium Rotary Instruments

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

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Abstract

BACKGROUND: Contemporary nickel-titanium (NiTi) rotary endodontic instruments had a revolutionary impact on the success of root canal treatment.

AIM: To evaluate the flexibility, microstructure and elemental analysis of four different recent NiTi rotary instruments, namely; Wave One Gold, TF adaptive, HyFlex EDM and Gr_Reciproc Blue compared to conventional Protaper Universal (F2).

MATERIAL AND METHODS: The NiTi rotary files were subjected to cantilever bending test to evaluate their flexibility. The microstructural characteristics and elemental analysis were examined via scanning electron microscopy (SEM) and energy dispersive X-ray spectrometer (EDX).

RESULTS: The TF adaptive, HyFlex EDM and Wave One Gold endodontic files showed significantly lower cantilever bending values (i.e., higher flexibility) than Protaper F2 and Gr_Reciproc Blue ($p < 0.05$). The SEM micrographs showed that the bulk of all examined files showed multiple striations due to the cutting process, on the other hand, their external surfaces were different: The Protaper Universal F2 showed multiple voids, while the TF Adaptive surface exhibited more uniform structure. The Hyflex EDM had a crater-like surface, whereas Wave one Gold showed machining grooves with minimum defects, while Reciproc Blue displayed machining grooves with random scratch lines. There was a significant difference in bulk and surface elemental analysis of the various examined files, yet composed mainly of the same elements.

CONCLUSION: Chemical composition, heat treatment, manufacturing process and geometrical design of the NiTi rotary instrument have a great influence on their flexibility and microstructure.

Introduction

Rotary Nickel-Titanium (NiTi) instruments were introduced to the endodontic field because of their superelastic behaviour over traditional stainless-steel instruments [1]. This made them the materials of choice for the shaping of curved root canals [1]. The unique superelasticity and shape memory effect of NiTi alloys is gained from their nearly equiatomic ratio. NiTi alloys of the rotary instruments consist of approximately 56% by weight nickel and 44% by weight titanium, which possess in 1:1 atomic ratio of the main components [2].

NiTi can be found in three different forms named; martensite, austenite and R-phase, which determine the mechanical properties and characteristics of the alloy [3]. Moreover, the mechanical performance of NiTi alloy depends on the relative characteristics of their microstructure and their association with the metallurgical behaviours, which is beneficial to understand the NiTi instruments behaviour [2].

Manufacturing of rotary NiTi endodontic instruments with different geometrical designs and chemical compositions have been developed [4]. However, sudden fracture of NiTi rotary files during root canal therapy remains a tremendous problem in the clinical aspect [4]. Thus, recently, NiTi endodontic files were fabricated by various thermal and mechanical treatment technologies to optimise their microstructure and the flexibility compared to the traditional superelastic NiTi files [4].

The flexibility of NiTi instruments means that NiTi files can permit a significant deformation below their elastic limits and still able to retain their original form [5], [6]. The flexibility of NiTi instruments play a crucial role in successful endodontic treatment because especially in curved canals, it permits suitable canal enlargement while preserving the instrument in a central position within the canal, leading minimum undesirable changes in the shape of curved canals [5], [6]. Flexibility is influenced by alloys chemical composition, heat treatment and geometric design such as file cross-sectional, and inner core area [7]. Low modulus of elasticity beside unique superelastic properties of NiTi endodontic instruments leads to superior flexibility [5], [6]. Flexibility evaluated usually in laboratory studies by cantilever bending test. The low bending result is indicative of the high material flexibility [7].

The metallurgical feature of NiTi instruments such as composition, microstructure and phase constitution has a great impact on their performance [8]. Metallurgical properties of NiTi instruments can be investigated by laboratory tests such as scanning electron microscopy (SEM) and energy dispersive X-ray spectrometer (EDX) [8].

Recent developments in the process of manufacturing of NiTi alloys are well documented, to produce contemporary rotary endodontic instruments with maximum flexibility [9]. New NiTi endodontic files with superior mechanical properties have been developed through special a series of thermo-mechanical processing treatment such as; producing NiTi alloys with the substantially stable martensite phase upon clinical conditions [9]. Alloys in its martensite form (M-Wire) become ductile, easily deformed and more flexible than austenite, thus reducing the risk of being broken under high stress [2].

Another manufacturing process is aiming to transform conventional NiTi alloy in the austenitic phase into a rhombohedral crystal structure (which called intermediate R-phase) between austenite and martensite. The rotary instrument made out of R-phase wire possesses good superelasticity, and lower rigidity than that of austenite, thus they exhibit more flexibility and can be twisted such as TF Adaptive files (TFA; SybronEndo, Orange, CA, USA) [3].

Hyflex EDM (HEDM; Coltene / Whaledent AG, Altstätten, Switzerland) was launched using electrical discharge machining (EDM) technique, which is a non-contact thermal erosion process via controlled electrical discharges machining process [10]. This process depends on electrical sparks which cause a local melting in addition to partial evaporation of small parts of metals leaving a typical crater-like surface finish. After that, the instrument is heat-treated at a temperature in a range of 300-600°C for 10 min-5 hours before or after the cleaning process [10]. The EDM process, being non-contact, is supposed to avoid early failure of material which may occur from conventional grinding techniques [4].

Wave One Gold endodontic file technology (Dentsply Maillefer, Ballaigues, Switzerland) is utilising a unique pre and post-manufacturing heat-treatment. The superelastic NiTi alloy is exposed to special heat treatment beneath constant strain in a range of 3-15 kg over a temperature range of 410°C-440°C. The finished instrument after machining is subjected to a further heat-treated in a range of 120°C-260°C. The manufacturer assumes that gold technology exhibited improved flexibility. They have two cutting edges; cross-section of the file was modified to a parallelogram [3].

Gr Reciproc Blue [RPC Blue; VDW, Munich, Germany] is released recently using innovative heat treatment that transforms the molecular structure of the M-Wire instrument providing a more flexible alloy; this treatment was also responsible for the blue colour of the file. They have two cutting edges; S-shaped cross-section and non-cutting tip [11].

Manufacturers have launched to market various thermally treated NiTi alloys (i.e., TF adaptive, HyFlex EDM) to optimise their microstructure and transformation, which in turn has a greater impact on their mechanical properties. The recent generation of NiTi instruments (i.e., Wave One Gold and Reciproc Blue subjected to a complex proprietary heating-cooling treatment providing a visible titanium oxide layer at the surface.

All this treatment and modification claimed by manufacturers to induce superior flexibility and high performance of the NiTi instruments. Thus, this study aims to evaluate the influence of NiTi alloy treatment of some recent NiTi rotary Instruments upon their bending behaviour compared to non-treated NiTi alloy (Protaper Universal, F2 [PTU; Dentsply Maillefer, Ballaigues, Switzerland]), in addition to the description of their microstructure and elemental analysis.

Material and Methods

Five different brands of rotary NiTi instruments (endodontic files) were tested in this study, Table 1: A) ProTaper Universal F2 (Dentsply Maillefer, Ballaigues, Switzerland); B) TF adaptive (SybronEndo, Orange, CA, USA); C) HyFlex EDM (Coltene / Whaledent AG, Altstätten, Switzerland); D) Wave One Gold (Dentsply Maillefer, Ballaigues, Switzerland); and E) Reciproc Blue (RPC Blue; VDW, Munich, Germany), (Figure 1).

Table 1: The endodontic rotary NiTi instruments tested

Brand Name / Taper	Manufacturer	ISO No. / Taper	Lot No.
Protaper Universal	Dentsply Maillefer, Ballaigues, Switzerland	25	1327775
TF Adaptive	SybronEndo Orange, CA, USA	25	051638929
Hyflex EDM	Coltene / Whaledent, Alstatten, Switzerland	25	H34710
WaveOne Gold	Dentsply Maillefer, Ballaigues, Switzerland	25	1332683
Reciproc Blue	VDW, Munich, Germany	25	041332

ProTaper Universal F2 file was established as the control group. All instruments tested with an ISO size 25 mm length with 0.40 mm tip.

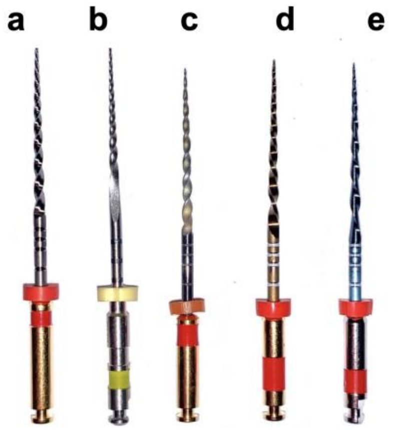

Figure 1: Rotary NiTi instruments: A) ProTaper Universal F2; B) TF adaptive; C) HyFlex EDM; D) Wave One Gold; E) Reciproc Blue

Flexibility (cantilever bending test)

These five different brands of rotary NiTi endodontic files were tested and divided into five experimental groups (n = 5). The rotary NiTi instruments' flexibility was evaluated by exposing the files to a cantilever bending resistance test using a universal testing machine (Model 3345; Instron Industrial Products, Norwood, MA, USA) with a load-cell of 5 kN. Each NiTi file was gripped horizontally in the lower fixed compartment of the universal testing machine by tightening screws. The tips of the specimens were subjected to a 45° displacement using mono-bevelled chisel attached to the upper movable compartment of the testing machine travelling at a cross-head speed of 0.5 mm/min. The chisel hit the file 5mm from its tip. Data were recorded using computer software (Instron® Bluehill Lite Software).

SEM and EDX analysis

Observations of the rotary NiTi instruments were conducted with a scanning electron microscope (JSM-5200, JEOL, Tokyo, Japan) equipped with EDX (Oxford Inca Energy 350, Oxford Instruments, Abingdon, UK) (n = 5). Specimens were prepared by cross-section cutting of the new files, then polishing it by silicon carbide sandpaper disc (400 to 2500 grit), under continuous running water. The chemical etching was done to reveal the metallographic features. The polished specimens were chemically etched in a solution of composed of 10 mL hydrofluoric acid, 45 mL nitric acid and 45 mL water and swabbed for 30 seconds according to ASTM E407-07 (Standard Practice for Micro-Etching Metals and Alloys) [12]. The analysis was performed along the cross-sectional area of the files; 5 mm from the tip of the file. Micrographs were taken at magnifications 400 X and 8000 X. The SEM micrographs and the EDX analysis were performed for both the bulk area and external surface of the different files to identify the microstructure and overall chemical composition respectively.

Statistical analysis

Statistical analysis of the data was conducted by using the One-way Analysis of Variance (ANOVA) and Tukey HSD test. The significance level was set to 5% ($P < 0.05$). Data obtained were analysed using IBM® SPSS® Statistics Version 20 for Windows (SPSS Inc., IBM Corporation; USA).

Results

Flexibility (cantilever bending)

The mean and standard deviation of the flexibility, measured by the load required to bend the NiTi instruments to 45°, are shown in Table 2.

Table 2: Mean and standard deviation of cantilever bending

Endodontic file	ProTaper Universal F2		TF adaptive		HyFlex EDM		Wave One Gold		Reciproc Blue		P-value
Cantilever bending (gram)	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	
	171.4^b	19.5	86.5^a	9.1	100.6^a	9	66^a	14.4	154.7^b	10.5	≤ 0.001*

Mean with different small letters indicate significance difference * statistically; significant ($p < 0.05$).

The three endodontic files; TF adaptive, HyFlex EDM and Wave One Gold showed significantly lower cantilever bending values (i.e. higher flexibility) than Protaper F2 and Gr_Reciproc Blue ($p < 0.05$).

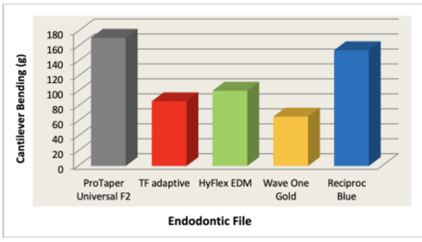

Figure 2: Bar chart showing mean cantilever bending values among the different endodontic files

SEM and EDX analysis

The SEM micrograph of Protaper Universal F2 displayed a convex triangular cross-section, Figure 3A. The bulk region showed multiple striations due to the cutting process, Figure 3B. This feature was not limited to the Protaper Universal F2 only, but all the tested endodontic files in this study. Where TF Adaptive, Hyflex EDM, Wave one Gold and Reciproc Blue showed nearly similar striations after cutting, Figures 4B, 5B, 6B and 7B respectively. Meanwhile, the external surface of Protaper Universal F2 showed multiple voids of varying sizes, Figure 3C.

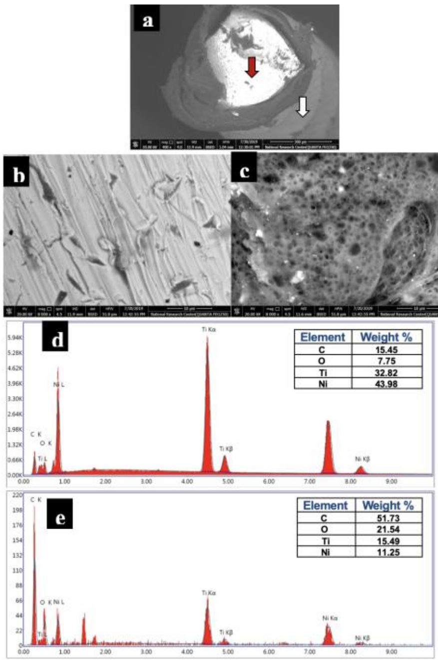

Figure 3: Representative SEM micrograph and EDX spectrum from a Protaper Universal F2 instrument; A) SEM micrograph of file cross-section after cutting, red arrow pointed to cut part of bulk and white arrow directed to external surface, magnification 400 X; B) SEM micrograph of cut part of bulk, magnification 8000 X; C) SEM micrograph of external surface, magnification 8000 X; D) EDX of cut part of bulk; E) EDX of external surface

Regarding TF Adaptive, its SEM micrograph revealed a triangular cross-section, Figure 4A. Contrary to the Protaper Universal F2, the external surface of TF Adaptive exhibited uniform structure with negligible voids, Figure 4C.

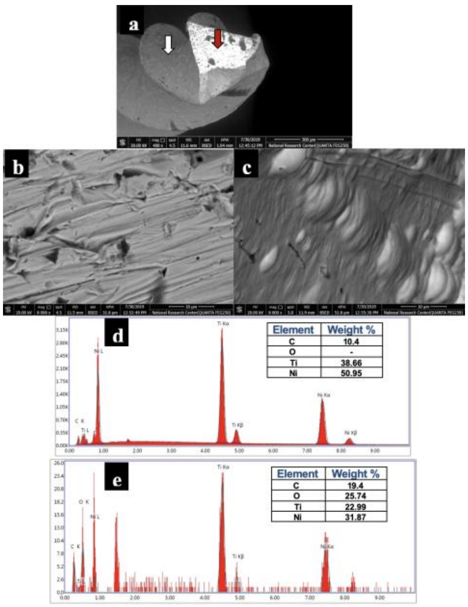

Figure 4: Representative SEM micrograph and EDX spectrum from a TF Adaptive instrument; A) SEM micrograph of file cross-section after cutting, red arrow pointed to cut part of bulk and white arrow directed to external surface, magnification 400 X; B) SEM micrograph of cut part of bulk, magnification 8000 X; C) SEM micrograph of external surface, magnification 8000 X; D) EDX of cut part of bulk; E) EDX of external surface

While, Hyflex EDM endodontic file had a rectangular cross-section, Figure 5A. Its external surface showed numerous corrugations with multiple pits and pores giving a crater-like surface, Figure 5C.

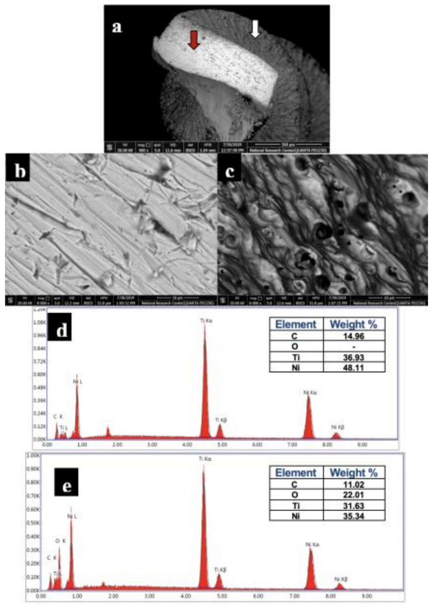

Figure 5: Representative SEM micrograph and EDX spectrum from a Hyflex EDM instrument; A) SEM micrograph of file cross-section after cutting, red arrow pointed to cut part of bulk and white arrow directed to external surface, magnification 400 X; B) SEM micrograph of cut part of bulk, magnification 8000 X; C) SEM micrograph of external surface, magnification 8000 X; D) EDX of cut part of bulk; E) EDX of external surface

The Wave one Gold showed parallelogram-shaped cross-section, Figure 6A. The SEM micrographs of its external surface-displayed machining grooves with minimum defects, Figure 6C.

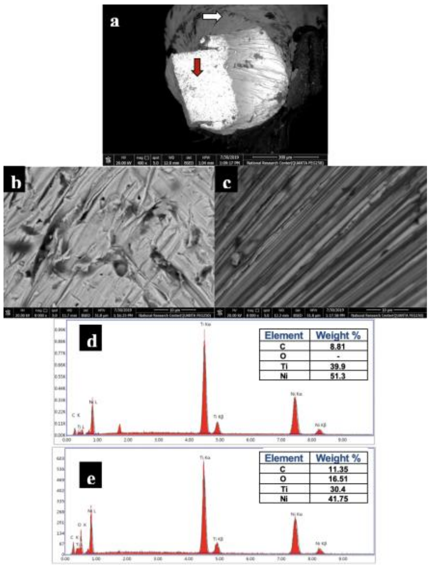

Figure 6: Representative SEM micrograph and EDX spectrum from a Wave one Gold instrument; A) SEM micrograph of file cross-section after cutting, red arrow pointed to cut part of bulk and white arrow directed to external surface, magnification 400 X; B) SEM micrograph of cut part of bulk, magnification 8000 X; C) SEM micrograph of external surface, magnification 8000 X; D) EDX of cut part of bulk; E) EDX of external surface

Concerning Reciproc Blue, its cross-section was dim S-shaped, Figure 7A. As for its external surface, machining grooves were shown with random scratch lines, Figure 7C.

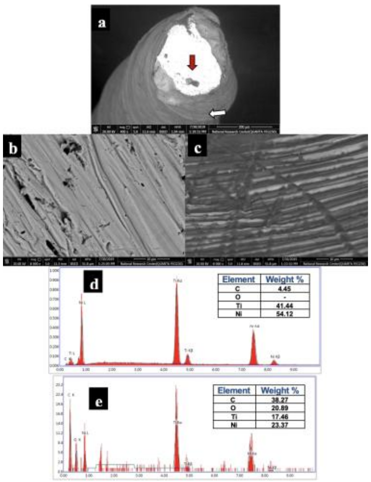

Figure 7: Representative SEM micrograph and EDX spectrum from a Reciproc Blue instrument; A) SEM micrograph of file cross-section after cutting, red arrow pointed to cut part of bulk and white arrow directed to external surface, magnification 400 X; B) SEM micrograph of cut part of bulk, magnification 8000 X; C) SEM micrograph of external surface, magnification 8000 X; D) EDX of cut part of bulk; E) EDX of external surface

EDX analysis showed that the bulk of all the examined five files were composed mainly of nickel and titanium, in addition to carbon in lower weight %, with a significant difference in these three elements among the different brands ($p < 0.05$), Table 3 and Figure 8.

Table 3: Mean and standard deviation of elemental analysis (EDX) of bulk material of the different endodontic files

Endodontic file	ProTaper Universal F2		TF adaptive		HyFlex EDM		Wave One Gold		Reciproc Blue		P-value
Elements (wt%)	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	
Carbon	15.5[e]	0.4	10.4[c]	0.4	15[d]	0.5	8.8[b]	0.2	4.5[a]	0.3	
Oxygen	7.8		-	-	-	-	-	-	-	-	≤ 0.001*
Titanium	32.8[a]	0.6	38.7[c]	0.4	36.9[b]	0.7	39.9[d]	0.3	41.4[e]	0.5	
Nickel	44[a]	0.3	51[c]	0.9	48.1[b]	0.5	51.3[d]	0.3	54.1[e]	0.6	

Mean with different small letters in the same row indicate significance difference * statistically; significant ($p < 0.05$).

Representative EDX spectra of bulk material of the different file brands are shown in Figures 3D, 4D, 5D, 6D and 7D. Oxygen was present in the bulk material of Protaper Universal F2 only, yet in very low percentage, Figure 3D. On the other hand, the oxygen was absent in the rest of the inspected files.

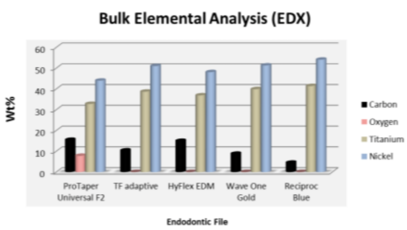

Figure 8: Bar chart showing elemental analysis (EDX) of bulk material of the different endodontic files

The surface EDX analyses of the different files are shown in Table 4 and Figure 9. There was a significant difference in external surface elemental analysis of various examined files, ($p < 0.05$) Representative EDX spectra of the surface of the different brands are shown in Figures 3E, 4E, 5E, 6E and 7E.

Table 4: Mean and standard deviation of elemental analysis (EDX) of the external surface of the different endodontic files

Endodontic file	ProTaper Universal F2		TF adaptive		HyFlex EDM		Wave One Gold		Reciproc Blue		P-value
Elements (wt%)	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	
Carbon	51.7[e]	0.6	19.4[c]	0.2	11[a]	0.6	11.4[b]	0.3	38.3[d]	0.5	
Oxygen	21.5[c]	0.4	25.7[e]	0.5	22[d]	0.5	16.5[a]	0.4	20.9[b]	0.7	≤ 0.001*
Titanium	15.5[a]	0.3	23[c]	0.8	31.6[e]	0.4	30.4[d]	0.3	17.5[b]	0.5	
Nickel	11.3[a]	0.2	31.9[c]	0.7	35.3[d]	0.3	41.8[e]	0.3	23.4[b]	0.7	

Mean with different small letters in the same row indicate the statistically significant difference, *; significant ($p < 0.05$).

Contrary to the bulk, the EDX analysis of the external surfaces of most tested files revealed that the carbon and oxygen were present in higher weight% than their percentages in the bulk.

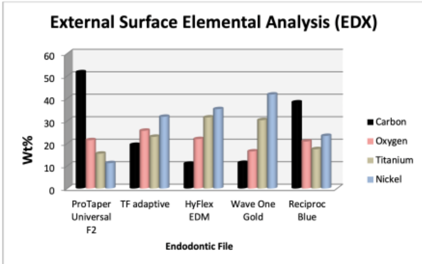

Figure 9: Bar chart showing elemental analysis (EDX) of external surfaces of the different endodontic files

Discussion

Using NiTi alloy for the production of endodontic instruments has brought pronounced advancement to endodontic treatment [13]. In this study, four rotary NiTi instruments were selected; TF adaptive, HyFlex EDM, Wave One Gold and Reciproc Blue. This selection was based on that they were subjected to different heat treatments and manufacturing process. Conventional Protaper Universal (F2) was used as a gold standard file [14].

Since the flexibility of NiTi files is considered an essential requirement in endodontic treatment, especially in curved canals [15], the flexibility of the examined file was compared in this research. It is usually assessed laboratory using bending test resembling the load exerted by the root canal curvature [7]. Lower values required for bending indicate higher flexibility of material [7].

In this study, Protaper F2 and Reciproc Blue endodontic files showed lower flexibility (higher cantilever bending values) compared to TF adaptive, HyFlex EDM and Wave One Gold. This might be attributed to several causes as the flexibility is a multifactorial property influenced by several variables as geometric conformation, chemical composition and heat treatment [7].

The cross-sectional configuration of Protaper F2 and Reciproc Blue and their elemental composition may play a role in decreasing their flexibility. It was stated previously that the cross-section geometry of Protaper Universal F2 (convex triangle) decreased their flexibility [16]. While, a previous study compared the cross-sectional area of different files (Reciproc Blue, WaveOne Gold and Genius File). It was found that the Reciproc Blue had the largest cross-sectional area (S-shape) [17]. It was reported previously that flat sides of endodontic files reduce the cross-sectional diameter and area, rendering the endodontic file thinner and more flexible to explore curved canals [18]. Thus, the S-shaped cross-section of Reciproc Blue and the convex triangle of Protaper Universal F2 with their non-flat sides may provide bulky cross-section area which decreases their flexibility.

On the other hand, the flat sides of TF adaptive, HyFlex EDM and Wave One Gold might play a role in increasing their flexibility as their cross-sectional design were triangle, rectangular and parallelogram respectively. The later cross-section was attributed to the increased flexibility of endodontic files [19]. From a mathematical point of view, there are certain shapes which have a lower cross-sectional area as a triangle and slender rectangle compared to square and rectangle [20]. Similarly, triangle with linear sides compared to a triangle with convex sides [21].

Another cause which might lead to lower flexibility of Protaper F2 and Reciproc Blue endodontic files compared to TF adaptive, HyFlex EDM and Wave One Gold was the percentage of carbon content in the surface. In this research, Protaper F2 surface had the highest carbon content (51.7wt%), followed by Reciproc Blue (38.3 wt%), then TF adaptive (19.4 wt%), HyFlex EDM (11 wt%) and Wave One Gold (11.4 wt%). The increased carbon on the surface may lead to more carbide precipitations which may obstruct dislocation movement, increasing file rigidity of Protaper F2 and Reciproc Blue [22], [23].

It should also be noted that the manufacturing process and heat treatment play a tremendous role in determining material flexibility [24]. Files which are produced by just milling without heat treatment as Protaper F2 displayed lower flexibility [24]. While those exposed to heat treatment showed more flexibility as the crystal structure and phase transformation temperatures are affected [4]. The manufacturing process which transforms the conventional NiTi alloy with austenitic phase into R-phase with rhombohedral crystal structure increases the flexibility of NiTi alloy. Thus, it can be used for the production of twisted files such as TF Adaptive files which have high flexibility [3]. While Wave one Gold is exposed to heat treatment before and after file machining [9]. It was documented that heat treatment of file after machining overcome the machining procedure defects and modify the crystalline phase with a significant increase in flexibility [25]. As for Hyflex EDM, it is fabricated by electrical discharge machining (EDM) followed by heat treatment which exhibited considerable amounts of the R-phase and martensite which increases flexibility [26]. Although Gr Reciproc Blue is produced by pre and post milling heat treatments, yet they show lower flexibility. This might be attributed to the other factors which may hinder flexibility as bulky cross-sectional area and increased carbon in surface [18], [22].

Heat treatment of NiTi alloys does not affect the mechanical properties of the endodontic files only but also their optical properties. When the instruments are heat-treated and then cooled, this results in a colour of surface corresponding to the thickness of titanium oxide layer [15]. If the titanium oxide layer thickness is 60-80 nm, this results in blue colour, while if the thickness is 100-140 nm, this results in golden colour [15]. This is confirmed in this research by EDX which analysed only Ti, Ni, O and C in the surface of these coloured files. Therefore, the term gold used in describing some endodontic files does not denote the presence of gold element but denote the gold colour of file and the heat-treatment performed in the file which imitates that done for gold alloy by heating and then slowly cooling after the production phase [27], [28].

Regarding the SEM micrographs, the bulk of all the examined files showed the same morphology (multiple striations) after cutting due to the standardisation of the cutting procedure. However, the external microstructure differs among the various files, which may be due to the different manufacturing process used for each.

The external surface of Protaper Universal F2 showed multiple voids of varying sizes. These voids were observed in previous studies examining Protaper files and attributed this to Kirkendall effect which occurs due to unbalanced atomic diffusion between nickel and titanium atoms due to the difference in their diffusion rates when heated which leads to the formation of voids [29], [30]. The nickel atoms diffuse faster into titanium than the titanium atoms do in the reverse direction. Thus the mass transport is not balanced which leads to voids formation in the nickel side after alloying [30]. Others attributed this void to inclusions as oxides impurities which entered within alloy during the manufacturing process. And after machining the files, these voids may lead to further machining grooves and cracks [30]. The presence of oxygen within the alloy of Protaper file was confirmed by EDX analysis in this study which detected oxygen in bulk material and may be reflected as voids in surface [30]. On the other hand, obvious voids were not detected in the surfaces of the other examined files nor oxygen was identified in their bulk materials. This might be attributed to the heat treatment processes performed in these files, as it was proven previously that heat treatments suppressed the formation of Kirkendall voids [31].

Contrary to the Protaper Universal F2, the surface of TF Adaptive exhibited uniform structure with negligible voids. This reason behind its great integrity with minimal defects may be due to the difference in its manufacturing process, as discussed previously. Its core structure is never sectioned but twisted which helps to preserve grain structure.

While the crater-like surface of Hyflex EDM may result from the electrical spark machining. As its surface showed numerous corrugations with a non-uniform structure where multiple pits and pores were observed as the electrical sparks cause local melting and partial evaporation of small parts of metals [32].

The external surface of Wave one Gold displayed even machining grooves without distortion with a uniform structure and minimum defects. This might be attributed to unique pre and post-manufacturing heat-treatment. It was stated previously that heat treatment of NiTi alloy led to a better crystalline arrangement which increases flexibility [33].

The Reciproc Blue surface showed machining grooves with random scratch lines; this might be attributed to its reported reduced micro-hardness compared to conventional M-Wire super-elastic NiTi [34]. Yet, it should be noted that hardness is a surface property and is not correlated to bulk properties as flexibility [35].

The EDX analysis results in this study are presented in weight percent for two mean reasons; first: in metallurgy, the weight percent is commonly used as it is used for determining how many grams would be added from each metal for alloy formation[36], second: for comparative reason as most previous researches used weight percent in describing their EDX results [37], [38], [39], [40].

The EDX analyses of the bulk material of the different files show that all of them consisted mainly of nickel and titanium [37]. Yet there was a significant difference in these elements among the different brands. This might be attributed to the discrepancies in the raw materials during manufacturing [38]. Carbon was also present in lower percentage as melting nickel and titanium was performed frequently using vacuum induction in graphite crucible [41], [42]. This procedure might lead to carbon contamination within the alloy [30], [42].

Contrary to the bulk, the EDX analysis of the surfaces of most tested files revealed that the carbon and oxygen were present in higher weight % than their percentages in the bulk. This may be due to the contact between the surface of NiTi endodontic file and the atmosphere which contains oxygen and carbon [43]. Thus, their concentration in the endodontic file increased from the surface and decreased towards the bulk [43]. Another justification of the increased carbon content in the surface was the decomposition of the oil used for lubrication during the machining process [44]. The oxygen on the NiTi surface is presented mainly as a titanium oxide layer which is responsible for tarnish and corrosion resistance and high biocompatibility of this material [45].

Within the limitations of the current study, it could be concluded that:

1. Chemical composition, heat treatment, manufacturing process and geometrical design of the NiTi rotary instrument have a great effect on their flexibility and microstructure.

2. Carbon and oxygen elements in the NiTi rotary instrument alloy affect their rigidity and structural integrity, respectively.

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