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## Influence of heat-treatment on torsional resistance to fracture of nickel-titanium endodontic instruments.

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### Abstract

Over the past 3 decades, Nickel-Titanium (NiTi) instruments have become an important part of the armamentarium for shaping phase of root canal treatment. NiTi endodontic files have increased flexibility and strength compared with stainless steel instruments, but they seem to be vulnerable to fracture in clinical situations. Many variables might contribute to file separation, but the 2 main causes are cyclic fatigue and torsional stress. Heat treatment (thermal processing) is one of the most fundamental approaches toward adjusting the transition temperatures of NiTi alloys and affecting the fatigue and torsional resistance of NiTi endodontic files. In recent years, novel thermo-mechanical processing and manufacturing technologies such as controlled memory wire (CM-wire), M-Wire and electrical discharge machining (EDM) have been developed to optimize the microstructure of NiTi alloys and their mechanical properties. Aim of this work was to investigate the torsional resistance (maximum torque load, and angular rotation) of NiTi instruments made by different thermo-mechanical and manufacturing processes.

One-hundred new Hyflex EDM One- File (#25/0.08, CM-wire and EDM process), WaveOne Primary (#25/0.08, M-wire), ProTaper Next X2 (#25/0.06, M-wire), Hyflex CM (#25/0.06, CM-wire) and F6 SkyTaper(#25/0.06, conventional NiTi) files were used. Torque and angle of rotation at failure of new instruments (n = 20) were measured using a torsionmeter according to ISO 3630-1 for each brand. Data were analyzed using the analysis of variance test and the Student- Newman-Keuls test for multiple comparisons. The fracture surface of each fragment was examined with a scanning electron microscope.

Files made by CM wire size #25, 0.06 taper (Hyflex CM) showed same torque load and angular rotation to fracture than conventional NiTi (F6 SkyTaper) ( $P > .05$ ); instead CM files (manufacturing by grinding or EDM process) recorded lower maximum torque load ( $P < .05$ ) but significantly higher angular rotation ( $P < .0001$ ) to fracture than M-wire for both instruments size #25, 0.06 taper and size #25, 0.08 taper (Hyflex EDM OneFile/WaveOne Primary; Hyflex CM/ProTaper Next X2). Conventional (F6 SkyTaper) NiTi files showed same torque load ( $P > .05$ ) but significantly higher angular rotation ( $P < .05$ ) to fracture than M-wire instruments size #25, 0.06 taper (ProTaper Next).

Hyflex EDM One-File and Hyflex CM have same torque load and angular rotation to fracture than F6 SkyTaper due to the higher flexibility and cross-sectional area of CM files tested than conventional NiTi one.

Moreover CM files showed lower torque load and higher angular rotation to fracture than M-wire instruments due to the flexibility of CM alloy. M-wire instruments showed same torque load but significantly lower angular rotation than conventional NiTi files due to the same flexibility and higher cross-sectional area of the files tested.

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## 1. Introduction

Despite the increased flexibility and strength, compared with stainless steel instruments, Nickel – Titanium (NiTi) endodontic rotary instruments are vulnerable to fracture (Walia et al. 1988, Sattapan et al. 2000, Cheung 2009, Pedullà et al. 2013a). One study found that NiTi files fractured 7 times more often than stainless steel files (Iqbal et al. 2006). Other studies found a file fracture rate of approximately 5% in clinical practice (Alapati et al. 2005, Parashos et al. 2004). Many variables might contribute to file separation, but the 2 main causes are cyclic fatigue and torsional stress (Pedullà et al. 2015). Each has been defined (Yum et al. 2011, Bhagabati et al. 2012), and clinically, cyclic fatigue seems to be more prevalent in curved root canals, whereas torsional failure might happen even in a straight canal (Plotino et al. 2010).

Torsional failure is characterized by a maximum torsional load and angle of rotation. This last property reveals the capability of the file to twist before fracture (Elnaghy & Elsaka 2015). Because of this, file manufacturers have tried to develop new designs, manufacturing processes, and kinematics to minimize fracture occurrence and create easier and faster techniques that maintain the original canal shape with considerably less iatrogenic error (Peters 2004, Capar et al. 2014).

In recent years, novel thermo-mechanical processing and manufacturing technologies such as controlled memory wire (CM-wire), M-Wire and electrical discharge machining (EDM) have been developed to optimize the microstructure of NiTi alloys and their mechanical properties. (Gao et al. 2010, Shen et al. 2013)

The M-wire NiTi is subjected to thermo-mechanical processing (Alapati et al. 2009) resulting in a reported increased flexibility (Larsen et al. 2009), which could result in better access and preparation of curved canals. ProTaper Next (Dentsply Maillefer, Ballaigues, Switzerland) (PTN) and WaveOne (Dentsply Tulsa Dental Specialties, Tulsa, OK) (WO) are files composed of M-wire but differ in their designs (Topçuoğlu et al. 2016).

The CM-Wire (called by manufacturers “Controlled Memory”) has been reported to be subjected to thermo-mechanical process and, unlike conventional files that possess a stress-induced phase transformation, these files behave more like what is termed martensitic-active or shape memory in orthodontic literature (Brantley 2001). Therefore, these files are also so called “shape memory files”. In fact, files made by CM-wire do not rebound (Ninan & Berzins 2013) to original shape like conventional NiTi files. Hyflex CM (HCM) and Hyflex EDM (HEDM) (Coltene/Whaledent AG, Altstätten, Switzerland) are two endodontic instruments made by CM-wire, but using two different machining methods. The shape of Hyflex CM instruments is obtained by traditional grinding process of a CM-wire. Instead, the shape of new Hyflex EDM is due to an electrical discharge machining (EDM) process. (Pedullà et al. 2016)

EDM can be used to manufacture all types of conductive materials (eg, metals, alloys, graphite, ceramics, and so on) of any hardness with high precision (Pedullà et al. 2016). During this procedure, the shape of a work piece is changed by building a potential between the work piece and the tools. The sparks initiated in this process are melting and vaporizing the material of the work piece in its top layer (Payal et al. 2008).

The EDM process creates a rough and hard surface that could improve the cutting efficiency of these files (Pedullà et al. 2016, Payal et al. 2008).

F6 SkyTaper (Komet/Gebr. Brasseler, Lemgo, Germany) are brand new instruments made by conventional NiTi shaped by traditional grinding processes. (Dagna et al. 2015)

WO Primary has a tip size of 25 with a 0.08 taper that is constant in the apical 3mm of the instruments, but it is reduced in the middle and coronal portion of the working part of the instrument (Plotino et al. 2012); moreover WO instruments have a modified convex triangular cross section at the tip and a convex triangular cross section in the middle and coronal portion of the instrument (Wycoff & Berzins 2012, Ruddle 2012).

ProTaper Next is a sequence of rotary instruments that are designed with variable tapers and an off-centred rectangular cross section. The system includes 5 shaping instruments: X1 (size 17, 0.04 taper), X2 (size 25, 0.06 taper); optional instruments X3, X4 and X5 (size 30, 0.075 taper, size 40, 0.06 taper, size 50, 0.06 taper, respectively), which are used depending on the dimensions of the root canal (Van der Vyver & Scianamblo 2014). These instruments are manufactured from M-wire that has extended fatigue life beyond conventional NiTi alloy (Johnson et al. 2008)

Hyflex CM instruments have a constant taper and a triangular cross-section.

HEDM OneFile has a tip size of 25 with a 0.08 taper. The taper is a constant 0.08 in the apical 4 mm of the instruments but reduces progressively up to 0.04 in the coronal portion of the instrument. This new file has 3 different

cross-sectional zones over the entire length of the working part (rectangular in the apical part and 2 different trapezoidal cross sections in the middle and coronal part of the instrument working portion) to increase its fracture resistance and cutting efficiency (Pedullà et al. 2016).

F6 SkyTaper have a “S-shaped” cross-section and a constant taper (Dagna et al. 2015).

Few data are available in literature about the torsional resistance of endodontic NiTi files that were subjected to different heat-treatment and/or manufacturing process. Therefore, the aim of this work was to investigate the torsional resistance (maximum torque load, and angular rotation) of NiTi instruments made by different thermo-mechanical and manufacturing processes as WaveOne Primary, ProTaper Next, Hyflex CM, Hyflex EDM and F6 SkyTaper.

## 2. Materials & Methods

One-hundred endodontic NiTi rotary instruments (also so called files) from five endodontic systems (two size #25, 0.08 taper as HEDM OneFile and WO Primary and three #25, 0.06 taper as Hyflex CM, F6 Skytaper and Protaper Next X2 ) were used in this study. All files used were 25-mm long, with 20 instruments consumed in torsional resistance tests. Every instrument was inspected for defects or deformities before the experiment under a stereomicroscope (SZR- 10; Optika, Bergamo, Italy); none were discarded.

The torsional load was applied until fracture to estimate the mean ultimate torsional strength and angle of rotation of the instruments tested using a custom-made device produced following ISO 3630-1 (Pedullà et al. 2015). Each file was clamped at 3 mm from the tip using a chuck connected to a torque-sensing load cell; after which, the shaft of the file was fastened into an opposing chuck able to be rotated with a stepper motor. The HEDM, Hyflex CM, ProTaper Next and F6 SkyTaper shaft were rotated in the clockwise direction, whereas the WO Primary one was rotated in the counterclockwise direction at a speed of 2 revolutions per minute until file separation. The torque load (Ncm) and angular rotation (deg) were monitored continuously using a torsionmeter (Sabri Dental Enterprises, Downers Grove, IL) at room temperature (21°C), and the ultimate torsional strength and angle of rotation at failure were recorded.

The length of the fractured file tip was measured by using a digital microcaliper (Mitutoyo, Kawasaki, Japan).

The fracture surfaces of all fragments were examined under a scanning electron microscope (ZEISS Supra 35VP; Oberkochen, GmbH, Germany) to look for topographic features of the fractured instruments.

The data were first verified with the Kolmogorov-Smirnov test for the normality of the distribution and the Levene test for the homogeneity of variances. Thus, data were statistically evaluated by the analysis of variance test and the Student-Newman-Keuls test for multiple comparisons (Prism 5.0; GraphPad Software, Inc, La Jolla, CA) with the significance level established at 5% ( $P < .05$ ).

Table 1. Mean torque and angle of deflection of instruments #25, 0.08 taper: Hyflex EDM (CM-wire) and WaveOne Primary (M-wire).

| Instrument         | Torque (Ncm)      |                    |      |      | Angle of Rotation (deg) |                    |     |     |
|--------------------|-------------------|--------------------|------|------|-------------------------|--------------------|-----|-----|
|                    | Mean              | Standard Deviation | Min  | Max  | Mean                    | Standard Deviation | Min | Max |
| Hyflex EDM OneFile | 1.21 <sup>a</sup> | 0.04               | 1.21 | 1.42 | 554.20 <sup>c</sup>     | 80.40              | 435 | 750 |
| WaveOne Primary    | 1.68 <sup>b</sup> | 0.14               | 1.58 | 1.96 | 220.30 <sup>d</sup>     | 33.37              | 172 | 260 |

Table 2. Mean torque and angle of deflection of instruments #25, 0.06 taper: F6 SkyTaper (traditional NiTi), Hyflex CM (CM-wire), ProTaper Next (M-wire).

| Instrument                             | Torque (Ncm)        |                    |      |      | Angle of Rotation (deg) |                    |     |     |
|--|---------------------|--------------------|------|------|-------------------------|--------------------|-----|-----|
|  | Mean                | Standard Deviation | Min  | Max  | Mean                    | Standard Deviation | Min | Max |
| F6 SkyTaper size #25, 0.06 taper       | 0.96 <sup>a,b</sup> | 0.09               | 0.72 | 0.98 | 476.10 <sup>c</sup>     | 29.60              | 413 | 560 |
| Hyflex CM size #25, 0.06 taper         | 0.86 <sup>a</sup>   | 0.08               | 0.73 | 0.90 | 588.40 <sup>c</sup>     | 86.30              | 502 | 816 |
| ProTaper Next X2, size #25, 0.06 taper | 1.18 <sup>b</sup>   | 0.20               | 0.98 | 1.45 | 273.10 <sup>d</sup>     | 27.40              | 216 | 298 |

## 3. Results

The mean and standard deviations of the torque maximum load, and angle of rotation until fracture for instrument

size #25, 0.08 taper and size #25, 0.06 taper are presented in Table 1 and Table 2 respectively. Different superscript letters indicate statistic differences among groups ( $P < .05$ ). Different superscript letters indicate statistic differences among groups ( $P < .05$ ).

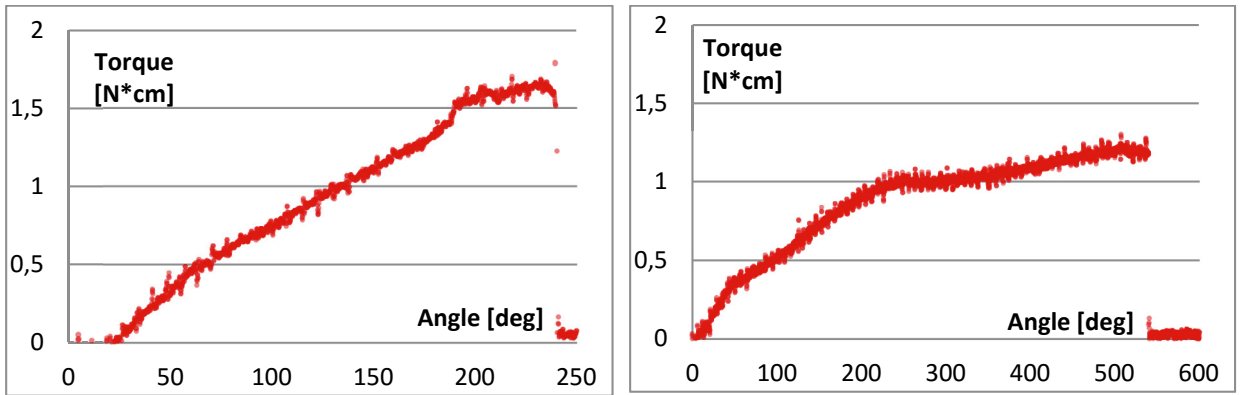


Figure 1. Torque vs. rotation curves for instruments with tip size 25 and 0.08 taper. Left: WaveOne Primary; right: Hyflex EDM OneFile.

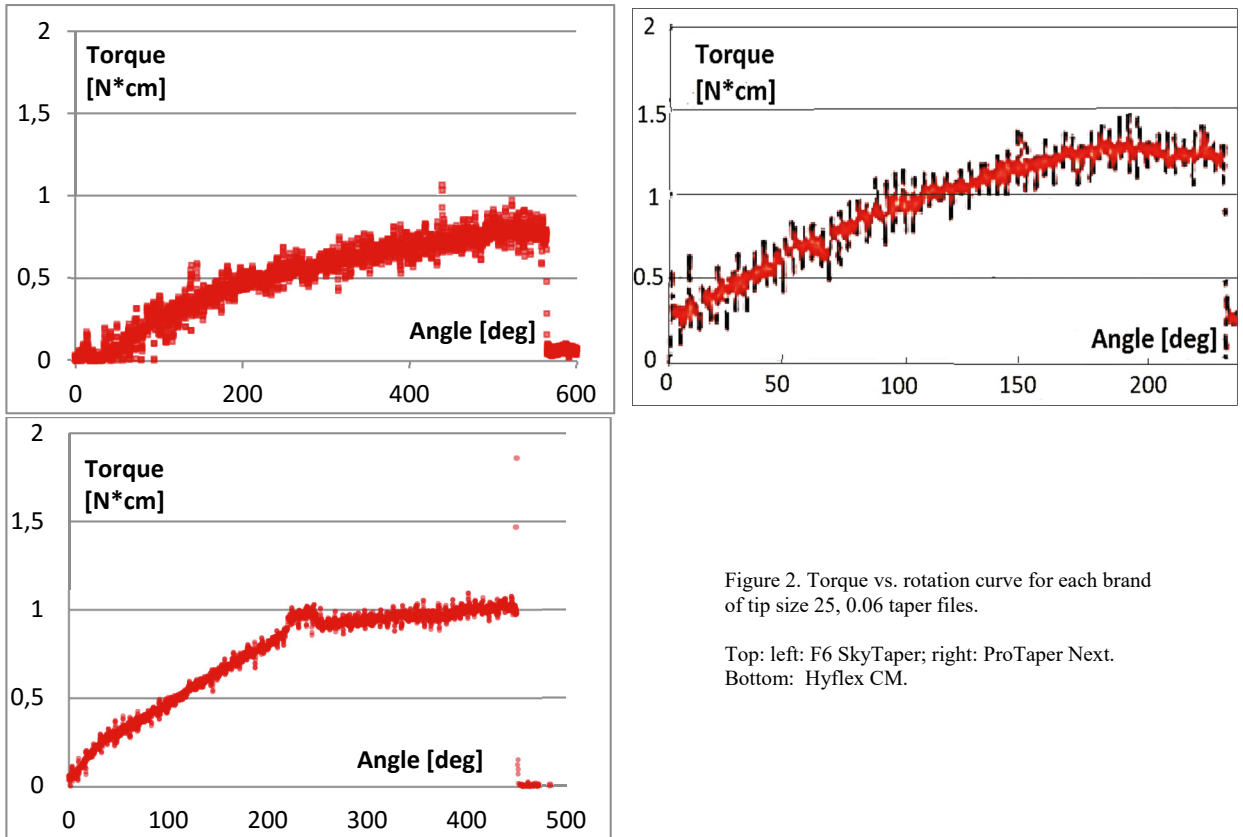


Figure 2. Torque vs. rotation curve for each brand of tip size 25, 0.06 taper files.

Top: left: F6 SkyTaper; right: ProTaper Next.  
Bottom: Hyflex CM.

Typical torque/angular rotation curves for torsional fracture for instrument size #25, 0.08 taper and size #25, 0.06 taper are presented in Figure 1 and Figure 2 respectively.

Between instrument size #25, 0.08 taper, HEDM OneFile (CM-wire) showed higher angular rotation ( $P < 0.0001$ ), but lower maximum torsional strength to fracture ( $P < 0.05$ ) than WO Primary (M-wire). In the same way, comparing instruments size #25, 0.06 taper, Hyflex CM (CM-wire) showed higher angular rotation ( $P < 0.0001$ ), but lower maximum torsional strength to fracture ( $P < 0.05$ ) than ProTaper Next X2 (M-wire).

F6 SkyTaper (conventional NiTi) showed same torque load and angular rotation to fracture than Hyflex CM (CM-

wire) ( $P > 0.05$ ), but same torque load ( $P > 0.05$ ) and higher angular rotation ( $P < 0.05$ ) than ProTaper Next X2 (M-wire).

The mean length of the fractured fragment (3.0 mm) was not significantly different for all of the instruments tested ( $P > 0.05$ ).

Scanning electron microscopy of the fracture surface showed similar and typical features of torsional failure for the 5 brands. The concentric abrasion marks and the fibrous dimple marks at the centre of rotation for torsional failure are shown in Figure 3 and 4.

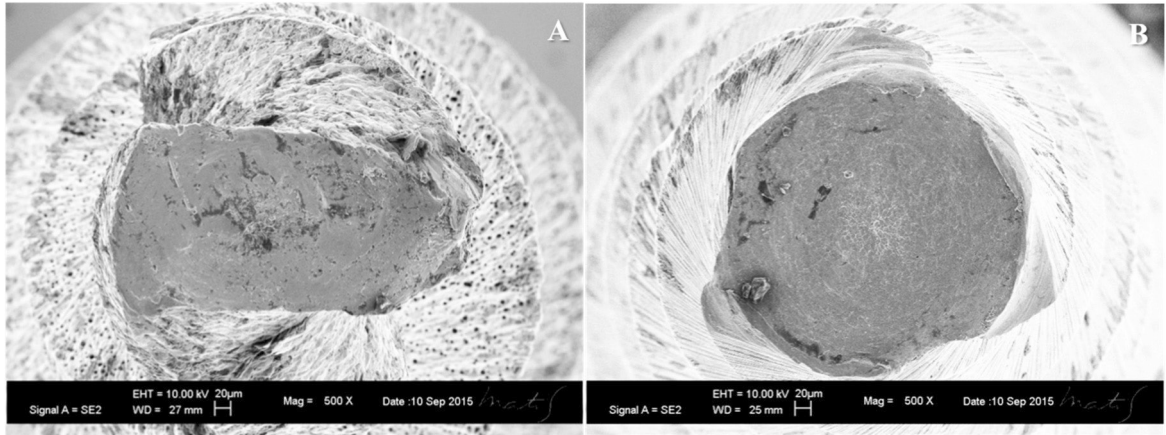


Figure 3 A-B. Scanning Electron Micrographs of fracture surface of separated fragments (A= Hyflex EDM OneFile; B = WaveOne Primary). (A–B) Concentric abrasion marks and skewed dimples near the center of rotation are typical features of torsional failure.

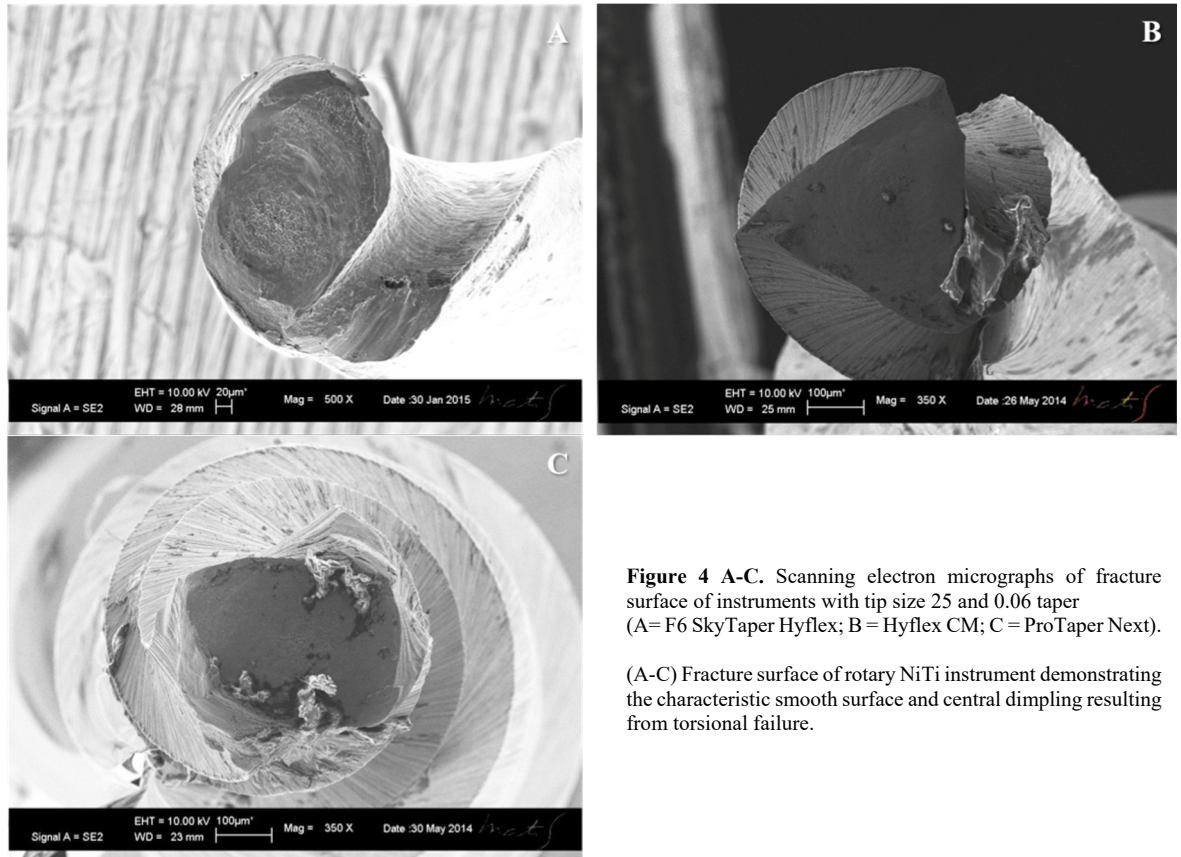


Figure 4 A-C. Scanning electron micrographs of fracture surface of instruments with tip size 25 and 0.06 taper (A= F6 SkyTaper Hyflex; B = Hyflex CM; C = ProTaper Next).

(A-C) Fracture surface of rotary NiTi instrument demonstrating the characteristic smooth surface and central dimpling resulting from torsional failure.

#### 4. Discussion

Several variables such as instrument size, taper, cross-sectional design, and manufacturing techniques affect the clinical performance of endodontic files and their resistance to fracture by torsion (Gao et al. 2010).

M-wire, the alloy used to manufacture WO and ProTaper Next, and CM-wire, the alloy used to make Hyflex CM and the new HEDM, are 2 types of heat-treated NiTi (Pedullà et al. 2016, Shen et al. 2013). Studies have investigated and compared the cyclic fatigue resistance of instruments made by these 2 types of heat-treated NiTi (Pedullà et al. 2015, Arias et al. 2014), however only few data are available about their torsional resistance to fracture.

In this research, torsional tests were performed following the ISO Standard 3630-1 as in previous studies (Bahia et al. 2008). The torque was applied in a counter-clockwise direction for WO and a clockwise direction for all other instruments tested because of the direction of their spiraling flutes (Kim et al. 2012). The broken fragment after the torsional tests showed an average length of 3 mm that coincided with the site of torsional loading application (at D3).

The files tested varied in cross-sectional designs and dimensions; thus, this report provides a comparison of specific files and not a systematic investigation of factors affecting file mechanical properties (Ninan & Berzins 2013).

In terms of cross-sectional design, at the level of torsional load apply (3mm from the tip), Hyflex EDM and Protaper Next are square, WaveOne and Hyflex CM are triangular and F6 SkyTaper is a “S-shaped” endodontic instrument. (Pedullà et al. 2015, Pedullà et al. 2016, Dagna et al. 2015).

As it was reported, there is a direct relationship between size of the file to torsional resistance. Similarly, the greater taper instruments display greater torque but less angle of rotation (Ninan & Berzins 2013). Therefore, data were compared between instruments that have same dimensions (tip size and taper).

For the #25 tip and 0.08 taper files, HEDM showed significantly higher angular rotation to fracture but a lower maximum torque load to failure than WO. In agreement with data reported in literature (Pedullà et al 2016), these results are probably caused by the different alloy and manufacturing processes of the instruments tested. In fact, in a supplementary examination, no significant differences were found in the cross-sectional area of the instruments tested (WO = 107587  $\mu\text{m}^2$  and HEDM = 110439  $\mu\text{m}^2$ ) measuring the cross-sectional configuration of each instrument captured at 3 mm from the tip (D3) under scanning electron microscopy by software (AutoCAD; Autodesk Inc, San Rafael, CA).

For the #25 tip and 0.06 taper files, Hyflex CM showed significantly higher angular rotation to fracture but a lower maximum torque load to failure than ProTaper NEXT X2. These results are probably due not only for the different alloy of the instruments tested, but also for the different cross-sectional area of these instruments (PTN = 118552  $\mu\text{m}^2$ , Hyflex CM = 98143  $\mu\text{m}^2$ ) (Pedullà et al. 2015). In agreement with these results, it was reported that M-wire instruments, such as WO and ProTaper Next, generally possess greater torque resistance but smaller angles of rotation before fracture than CM-wire files (such as HEDM and Hyflex CM) (Ninan & Berzins 2013, Shen et al. 2013). Moreover, as already reported, instruments with a big cross-sectional area should have higher torsional resistance than the ones with a small cross-sectional area (Schafer et al. 2003, Melo et al. 2008).

Among the instruments #25 tip and 0.06 taper files tested, F6 SkyTaper (conventional NiTi) showed same torque load and angular rotation to fracture than Hyflex CM (CM-wire) ( $P > 0.05$ ). These findings are probably due to the CM-wire (Hyflex CM) angular rotation and torque load resistance higher and lower respectively than conventional NiTi (F6 SkyTaper) as reported in literature from one hand and from the other hand due to the Hyflex CM cross-sectional area higher than the one of F6 SkyTaper (80548  $\mu\text{m}^2$ ) that cause its lower angular rotation and higher torque load resistance than F6 SkyTaper.

Moreover F6 SkyTaper showed same torque load than ProTaper Next X2 (M-wire). These results are probably due to the higher flexibility of M-wire than conventional NiTi compensated by the smaller cross-sectional area of F6 SkyTaper than the one of ProTaper Next (PTN = 118552  $\mu\text{m}^2$ ).

On the other hand, F6 SkyTaper showed higher angular rotation than ProTaper Next X2 (M-wire). These results are probably due to the higher impact of the cross-sectional area than crystalline alloy structure differences on flexibility, and therefore on angular rotation resistance, of ProTaper Next X2 (M-wire) than F6 SkyTaper (conventional NiTi). In fact, the cross-sectional area of F6 SkyTaper is really smaller (80548  $\mu\text{m}^2$ ) than the one of ProTaper Next (PTN = 118552  $\mu\text{m}^2$ ).

The SEM analysis revealed typical fractographic appearances of torsional fractures that were similar amongst the five brands tested. Torsional failure is characterized by circular abrasion marks and dimples near the centre of rotation on the fracture surface (Parashos & Messer 2006, Campbell et al. 2014).

The clinical implications of the high angle of rotation before fracture in CM-wire instruments tested may be beneficial because it may provide clinicians an indication that there is plastic/permanent deformation and fracture is imminent (Ninan & Berzins 2013). On the other hand, WO could be used for the constricted canal that might induce higher torsional load stresses (Kim et al. 2012); whilst F6 SkyTaper files have medium values of torsional resistance and it will be possible to use them in a high percentage of root canals.

## 5. Conclusions

Comparing instruments of the same dimensions, results showed higher flexibility and angular rotation to fracture but a lower maximum torque load to failure of HEDM and Hyflex CM (CM-wire for both files) than WaveOne and ProTaper Next (M-wire for both files) respectively. Moreover, EDM manufacturing process seems to be a potential production method of endodontic mechanical instruments.

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