Evaluation of physicochemical and mechanical properties of NiTi endodontic instruments made with conventional and controlled-memory alloys

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DOI: http://dx.doi.org/10.14436/2358-2545.6.2.028-033.oar

ABSTRACT

Objective: The aim of this study was to evaluate the semiquantitative composition, flexibility and microhardness of endodontic instruments with conventional and CM NiTi alloy, and to evaluate the influence of continuous rotation and reciprocating movement on the static fatigue life. **Methods:** Hyflex CM 40/0.04 and RaCe 40/0.04 were submitted to cantilever-bending test, energy dispersive X-ray spectroscopy and Vickers microhardness. Static fatigue tests were performed under continuous rotation; 150° clockwise/30° counterclockwise; 75° clockwise/15° counterclockwise. Results: Hyflex CM is more flexible than RaCe ($p < 0.05$) whereas no significant difference was observed regarding the microhardness ($p > 0.05$). RaCe has 55.7%wt Ni and 44.3%wt Ti, while Hyflex CM has 50.81%wt Ni and 42.28%wt Ti. Reciprocation motion showed statistically significant longer fatigue life when compared to the CR for both instruments ($p < 0.05$). Hyflex CM used with the reciprocating motion with the smallest angle of reciprocation showed statistically significantly longer fatigue life compared with the greater angle ($p < 0.05$) whereas there was no significant difference for the RaCe instruments ($p > 0.05$). Hyflex CM showed longer fatigue life than RaCe for all rotation movements ($p < 0.05$). **Conclusion:** Hyflex CM had higher flexibility and lower percent of Ni than RaCe. Both instruments had similar microhardness. Hyflex CM had longer fatigue life than RaCe in all rotation movements. Both files had longer fatigue life in reciprocating motion than continuous rotation. Smaller reciprocating angle had better fatigue resistance when compared to the greater angle.

Keywords: Endodontics. Fatigue. Titanium. Nickel. Rotation.

How to cite this article: Lopes HP, Vieira MVB, Elias CN, Villagra RM, Vieira VTL, Souza LC. Evaluation of physicochemical and mechanical properties of NiTi endodontic instruments made with conventional and controlled-memory alloys. Dental Press Endod. 2016 May-Aug;6(2):28-33.
DOI: http://dx.doi.org/10.14436/2358-2545.6.2.028-033.oar

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Submitted: 12/02/2016. Revised and accepted: 05/07/2016.

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Introduction

Since its introduction in Endodontics, the nickel-titanium (NiTi) instruments have become the instrument of choice to prepare curved root canals. These instruments have a lot of advantages, such as, improved flexibility and more rapid and centered root canal preparation.[1]

However, these instruments have a high risk of fracture during use in curved canals. To overcome this disadvantage, different manufacturing procedures have been proposed.[2,3,4] The flexibility and resistance to fatigue fracture of the NiTi endodontic instrument can be improved by different cross-section designs and alloys.[2,3,4] Thermomechanical treatments have been used to obtain new NiTi alloys, such as, R-phase, M-wire and controlled-memory alloy (CM), but the exact treatments are not disclosed by the manufactures.

The time to fatigue fracture can also be increased by subjecting the instrument to different rotation movements. The reciprocating movement was introduced by Yared[5] and it occurs when the instrument rotates in one direction and reverses direction before completing one cycle.[6] Several studies demonstrate that the reciprocation movement increases the fatigue life of the instrument when compared to the continuous rotation.[3,7-14]

The aims of this study were to evaluate the semiquantitative composition, flexibility and microhardness of endodontic instruments with conventional and CM NiTi alloy, as well as to evaluate the influence of continuous rotation and reciprocating movement on the fatigue life of these instruments subjected to static test.

Materials and Methods

The rotary NiTi instruments used in this study were Hyflex CM (Coltene-Waledent, Allstatten, Switzerland), an endodontic instrument made with control memory NiTi alloy and RaCe (FKG Dentaire SA, La Chaux-de-fonds, Switzerland), made with conventional NiTi alloy. The instruments selected had nominal size of 0.40 mm at D0, length of 25 mm and constant taper of 0.04 mm/mm along the working length.

Bending resistance test

The instruments' flexibility was evaluated by the cantilever-bending resistance test, as described in a previous study.[15] Briefly, a universal testing machine (Emic DL10.000, São José dos Pinhais, PR, Brazil) was used. And load was applied by means of a flexible stainless steel wire (diameter of 0.18 mm), with one of the extremities fastened to the testing machine head and the other end 3 mm away from the instrument tip. The bending test was conducted until the tip of each specimen underwent an elastic displacement of 45°. The test speed was 15 mm/min, and the load cell used was 20 N. Ten of each instrument was evaluated.

Data were statistically analyzed by using the Student's *t* test, with the significance level set at 5% ($P < 0.05$).

Vickers microhardness

Two instruments of each commercial brand were embedded in epoxy resin and to expose the instrument nucleus it was used sandpaper 200, 300, 400, 600 and 1200. Alumina with particles of 0.5 µm was used to polish the samples.

The Vickers microhardness was made with 100 g during 15 s using a microdurometer Bhueler model 1600-5300. Five penetrations were done in the central area of the work portion of each instrument.

Data were statistically analyzed by using the Student's *t* test, with the significance level set at 5% ($P < 0.05$).

Cyclic fatigue tests

An artificial canal was used for the fatigue tests, which was fabricated from a stainless steel tube, measuring 1.4 mm in diameter and 19 mm in total length. Between two straight segments, with 7 mm and 3 mm-long, it was created a 9 mm-long curved segment with 6 mm radius (measured at the internal concave surface of the tube). The canal was filled with glycerin to reduce friction, minimizing the release of heat.

These tests were conducted by using a mechanical apparatus previously described by Lopes et al.[15] The fatigue tests were performed under static condition. The instruments were driven by an engine (Satelec Endo Dual Motor; Acteon, Mérignac, France) at 300 rpm and with a 16:1 reduction handpiece (W&H, Dentalwerk, Bürmoos GMBH, Austria). Based on the kind of rotation movement used, the instruments were divided into the following groups:

» Group 1 – Continuous rotation (Hyflex CM – n=10 / RaCe – n =10).

» Group 2 – Reciprocation movement: 150° clockwise (CW) and 30° counterclockwise (CCW) (Hyflex CM – n=10 / RaCe – n =10).

» Group 3 – Reciprocation movement: 75° CW and 15° CCW (Hyflex CM – n=10 / RaCe – n =10).

The instruments were operated in the canal at a fixed length until fracture occurred. The time of fracture was recorded by the same evaluator by using a digital stopwatch (Technos, Manaus, Brazil) and was established when fracture was detected visually and/or audibly.

Data were statistically analyzed by intragroup and intergroup comparisons, with the significance level set at 5% ($p < 0.05$).

Intragroup comparisons of the cyclic fatigue resistance for each file with different rotation movements were analyzed by one-way ANOVA and Tukey test for multiple comparisons.

Intergroup comparisons of the cyclic fatigue resistance between each file for all different rotation movements were analyzed by Student's *t* test.

Scanning Electron microscopic (SEM) and Energy dispersive X-ray spectroscopy (EDS)

The cross section of both instruments were analyzed by scanning electron microscopic (SEM) (FEI QuantaTM 250 FEG - Field Emission Gun - FEI, Germany). Two instruments of each brand were embedded in acrylic resin and processed to be analyzed.

The semiquantitative composition of both instruments was done with an EDS Bruker e- Flash (FEI, Germany).

After the cyclic fatigue tests, the type of fracture of the surfaces and the presence of plastic deformation at the helical shaft were analyzed under SEM.

Results

Bending resistance test

The mean values and standard deviations for the bending resistance, measured by the maximum load (in grams) to bend the instruments, are shown in Table 1. A significant difference was observed between the instruments tested. Hyflex CM is significantly more flexible than RaCe ($p < 0.05$).

Vickers microhardness

The mean values and standard deviations for the microhardness are shown in Table 1. There was no statistically significant difference between the instruments ($p > 0.05$).

Cyclic fatigue tests

The results for the static fatigue test with different rotation movements are shown in Figure 1. The time to fracture under continuous rotation for Hyflex CM and RaCe were respectively 87.5 ± 11.34 and 56 ± 7.87 seconds. Under reciprocating motion of 150° CW and 30° CCW, the time to fracture were 647.3 ± 95.88 seconds for Hyflex CM and 144.3 ± 15.64 for RaCe. And for the motion with 75° CW and 15° CCW, the time was 765 ± 101.91 seconds for Hyflex CM and 154.4 ± 20.49 seconds for RaCe.

Regardless the angle of reciprocation, the reciprocation motion showed statistically significantly longer fatigue life when compared to the continuous rotation for both instruments ($p < 0.05$).

Hyflex CM used with the reciprocating motion with the smallest angle of reciprocation showed statistically significantly longer fatigue life compared with the greater angle ($p < 0.05$).

There was no significant difference between the reciprocating motion for the RaCe instruments ($p > 0.05$).

Hyflex CM showed longer fatigue life than RaCe for all rotation movements ($p < 0.05$).

SEM and EDS

Both instruments had triangular cross section, but Hyflex CM showed convex sides (Fig 2).

All instruments had morphologic characteristics of ductile fracture since it had the presence of microvoids into fracture surface and no plastic deformation was observed in the helical shaft of the instruments (Fig 3).

The EDS showed that major chemical components of both instruments are niquel and titanium. Small amounts of oxygen were found because of the oxide surface layer. RaCe has 55.7%wt Ni and 44.3%wt Ti, while Hyflex CM has 50.81%wt Ni and 42.28%wt Ti.

Table 1. Bending resistance and microhardness mean value

Instrument	Bending resistance (g)	Microhardness (VMH)
Hyflex CM	241 ± 19*	345 ± 14.42*
RaCe	403 ± 17**	348.5 ± 24*

Different superscript signals (*) represent statistically significative difference among the instruments ($p < 0{,}05$).

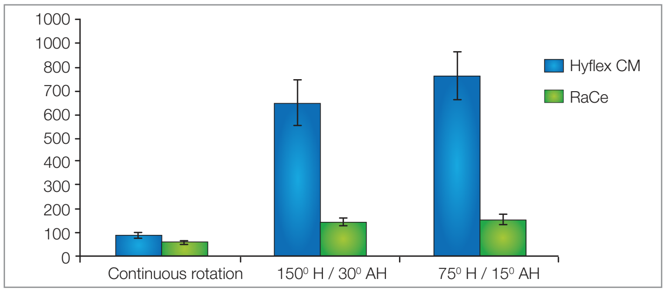

Figure 1. Graphic showing the mean and standard deviation time to fracture in seconds of each instrument under static cyclic fatigue test with continuous rotation and reciprocation movement of 150° clockwise (CW) and 30° counterclockwise (CCW), and 75° CW and 15° CCW. Intragroup comparison: different superscript letter represent statistical differences between rotation movements for each instrument (p<0.05). Intergroup comparison: different superscript sign (*) represent statistical differences between instruments for each rotation movement (p<0.05).

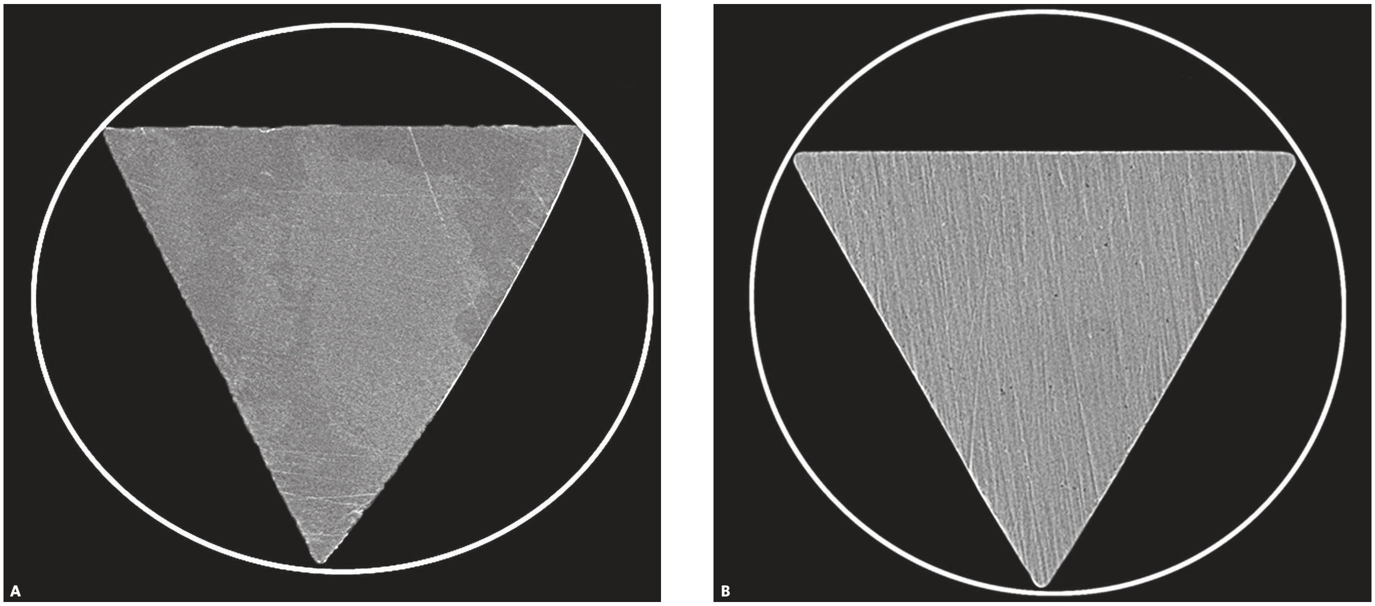

Figure 2. Cross section of the instruments tested. (A) Hyflex CM; (B) RaCe (original magnification, x100).

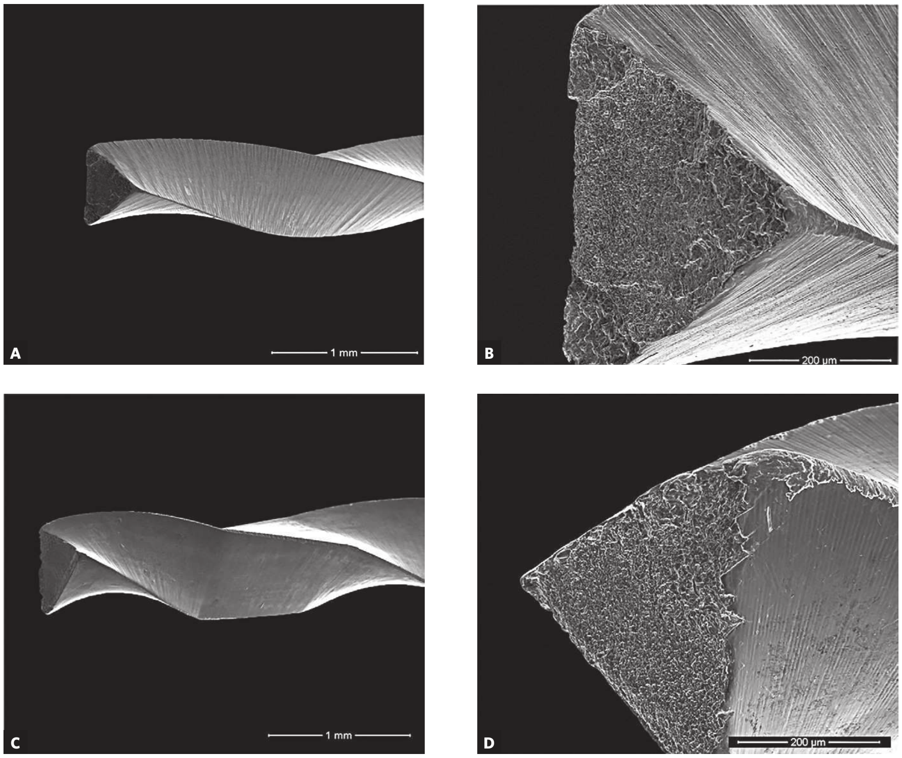

Figure 3. Fractured instruments. No plastic deformation is observed on the helical shaft. Fracture surfaces show morphologic characteristics of the ductile type: **A**) Hyflex CM files (original magnification, x100). **B**) Hyflex CM files (original magnification, x500). **C**) RaCe files (original magnification, x100). **D**) RaCe files (original magnification, x500).

Discussion

The instruments used in this study, Hyflex CM and RaCe, have similar cross-section design and the same nominal size of 0.40 mm at D0, length of 25 mm and constant taper of 0.04 mm/mm along the working length.

RaCe is manufactured with conventional NiTi alloy, whereas Hyflex CM is manufactured with NiTi control memory (CM) alloy. The NiTi alloy is subjected to thermomechanical process that originates the CM alloy.[16] This process makes the instruments extremely flexible because it controls the material's memory.[16] This alloy showed lower percent in weight of Ni (50.81 %wt) when compared to RaCe instruments (55.7 %wt). Zinelis et al.[17] compared the chemical composition of a manual Hyflex X-File with nine NiTi instruments (manual and rotary). The EDS analysis showed that the Hyflex X-File had 52.1%wt of Ni while the instruments had between 54.2 and 56.1%wt of Ni content.

Hyflex CM showed higher flexibility than RaCe. As observed in other studies, the control memory alloy has a major influence on the highest flexibility of the Hyflex CM when compared with file made of different alloys.[18,19]

The obtained microhardness results showed that there was no difference between the NiTi alloys. Lopes et al.[20] observed average values of 345 VMH in NiTi instruments, which was in accordance to our results.

In order to standardize the assay and to reduce the variables, the static fatigue test was conducted in an artificial canal made by a metallic tube with standardized length of the canal, length of the curvature radius and the length of the arc.[15,21] However, one should bear in mind that the actual lengths of the arc and the radius of the cylindrical curved canal are not the same as those of the instrument positioned inside the tube.[22] These discrepancies are due to the instrument's flexibility and to the relationship between the diameter of the instrument and the inner diameter of the canal, and may contribute to variations in the fatigue life of endodontic instruments tested in cylindrical canals.

In the present study, the fatigue life was evaluated by time[3,4] and not by the number of cycles to failure (NCF).[7,8] We opted for not considering the NCF to the resistance to fatigue fracture because the assessment of the NCF during reciprocating movement cannot be obtained by simply multiplying the rotation speed by the time elapsed until fatigue fracture. Instead, to determine the NCF in this situation it is necessary to know the amplitude (angle) of the oscillating motion in each cycle, as well as the frequency of oscillation within a constant time unit, which are parameters generally not provided by the manufactures. Time until failure represents more clinically relevant information, because time is much easier for the operator to control than the number of cycles the instrument endures.[6]

The static fatigue test was carried under continuous rotation and reciprocating movements with different angles to analyze the influence of rotation movements. Hyflex CM showed longer fatigue life than RaCe for all rotation movements. The flexibility of the instrument may be affected by the instrument design and dimensions, chemical composition of the alloy and thermomechanical processing.[18] In the present study, both instruments had the same nominal size (D0 and taper) and similar cross section design, therefore, the increased fatigue life of the Hyflex CM can probably be explained by the different alloys of both instruments.[18,23]

Regardless the type of instrument, the fatigue life was significantly lower when the instruments were driven under continuous rotation. These results are in agreement with several studies.[3,7-14] The rotary instrument under continuous rotation within a curved canal suffers alternating tensile and compressive stresses in a single point, whereas in the reciprocation motion there is better distribution of the alternating stresses. Consequently, the instrument operated under continuous rotation has shorter fatigue life.[9]

Since the instruments tested in this study have the helicoidal shaft from the right to the left, the instrument cuts to the right, so it has to turn more to the right (CW) to cut the dentin, and less to the left (CCW), to release the instrument from the dentin.

Hyflex CM used with the reciprocating motion with the smallest angle of reciprocation (75° CW / 15° CCW) showed statistically significantly longer fatigue life compared with the greater angle (150° CW / 30° CCW). The instruments under the reciprocation with the smallest angle need six CW and CCW movements to make one cycle. This means that the tensile stresses at the maximum flexural point are distributed to six points. The reciprocation movement with greater angle needs three CW and CCW movements to complete one cycle, therefore, the tensile stresses are distributed to three points. This can explain the longer fatigue life of the instruments subjected to the smallest angle of reciprocation.[3,13] Although not significant, RaCe also showed longer fatigue life when used under reciprocation movement with the smallest angle. Other studies, testing different instruments, also showed a shorter fatigue life when the instruments were used under reciprocation movement with the greater angle.[24,25]

SEM analysis revealed morphological characteristics of ductile fracture and no plastic deformation was detected in the helical shaft of the tested instruments. This is in accordance with previous studies.[9,14]

Conclusions

Based on the obtained results it can be concluded that Hyflex CM had higher flexibility and lower percent of Ni when compared to RaCe. Both instruments had similar microhardness. Hyflex CM had longer fatigue life than RaCe in all rotation movements. Both files had longer fatigue life in reciprocating motion than continuous rotation. Smaller reciprocating angle had better fatigue resistance when compared to the greater angle.

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