

## ***In vitro* evaluation of cyclic fatigue resistance of thermally treated novel nickel-titanium rotary instruments**

**Avaliação *in vitro* da resistência à fadiga cíclica de novos instrumentos rotatórios de níquel-titânio tratados termicamente**

**Evaluación *in vitro* de la resistencia a la fatiga cíclica de nuevos instrumentos rotatorios de níquel-titanio tratados térmicamente**

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**Bianca Reis Assaf**

ORCID: <https://orcid.org/0000-0003-2980-1146>  
Centro Universitário Newton Paiva, Brazil  
E-mail: [biancareisassaf@hotmail.com](mailto:biancareisassaf@hotmail.com)

**Júlia Carolina Verçosa**

ORCID: <https://orcid.org/0000-0003-0189-3667>  
Centro Universitário Newton Paiva, Brazil  
E-mail: [julia\\_carolina2@hotmail.com](mailto:julia_carolina2@hotmail.com)

**Vanessa Horrane da Silva Costa**

ORCID: <https://orcid.org/0000-0003-2000-2049>  
Centro Universitário Newton Paiva, Brazil  
E-mail: [vanessahorrane@hotmail.com](mailto:vanessahorrane@hotmail.com)

**Alice Machado Carvalho Santos**

ORCID: <https://orcid.org/0000-0002-6864-246X>  
Universidade Federal de Minas Gerais, Brazil  
E-mail: [alicemachado\\_santos@hotmail.com](mailto:alicemachado_santos@hotmail.com)

**Marcos Frozoni**

ORCID: <https://orcid.org/0000-0001-8001-4063>  
Faculdade de Odontologia São Leopoldo Mandic, Brazil  
E-mail: [marcosfrozoni@gmail.com](mailto:marcosfrozoni@gmail.com)

**Alexandre Moreira Fernandes**

ORCID: <https://orcid.org/0000-0001-6928-2024>  
Centro Universitário Newton Paiva, Brazil  
E-mail: [endoamf@gmail.com](mailto:endoamf@gmail.com)

### **Abstract**

The objective of this study was to evaluate the cyclic fatigue resistance of novel NiTi files (subjected to heat treatment) using the *in vitro* model (artificial canal). Twelve ProDesign Logic instruments - PDL 25/.06 (Easy Equipamentos Odontológicos, Belo Horizonte, Brazil) and 12 Protaper Next X2 instruments - PTN (tip 25) were included in this research. The Static cyclic fatigue test was performed with a grooved stainless steel block simulating a canal of 1,5 mm diameters, a 60° angle of curvature and 5 mm radius. The files were positioned inside the artificial canal and rotated until the fracture occurred. Using the time until fracture (seconds) and the number of rotations per minute (RPM), the number of cycles until fracture (NCF) was calculated and the length of ruptured fragments was registered. Three samples from each group were analyzed in Scanning Electronic Microscopy (SEM) to characterize the metal rupture. Data were analyzed using the Mann-Whitney non-parametric test and the level of significance considered was  $p < 0.05$ . PDL obtained a mean value of 956,0 NCF (352,0 - SD) and PTN achieved 391,0 NCF (36,7 - SD) with statistical significance between the groups ( $p < 0.001$ ). There was no statistical difference on fragment length ( $p > 0.05$ ). SEM analysis showed features compatible with ductile fracture. ProDesign LOGIC files showed higher NCF than Protaper Next X2. There was no difference between groups considering the length of the ruptured fragment.

**Keywords:** Dental instruments; Endodontics; Rotation.

### **Resumo**

O objetivo deste estudo foi avaliar a resistência à fadiga cíclica de novas limas de NiTi (sujeitas a tratamento térmico) usando o modelo *in vitro* (canal artificial). Doze instrumentos ProDesign Logic - PDL 25/.06 (Easy Equipamentos Odontológicos, Belo Horizonte, Brasil) e 12 instrumentos Protaper Next X2 - PTN (ponta 25) foram incluídos nesta pesquisa. O ensaio de fadiga cíclica estática foi realizado com um bloco de aço inoxidável ranhurado simulando um canal de 1,5 mm de diâmetro, ângulo de curvatura de 60° e raio de 5 mm. As limas foram posicionadas dentro do

canal artificial e giradas até ocorrer a fratura. Usando o tempo até a fratura (segundos) e o número de rotações por minuto (RPM), o número de ciclos até a fratura (NCF) foi calculado e o comprimento dos fragmentos rompidos foi registrado. Três amostras de cada grupo foram analisadas em Microscopia Eletrônica de Varredura (MEV) para caracterizar a ruptura do metal. Os dados foram analisados pelo teste não paramétrico de Mann-Whitney e o nível de significância considerado foi de  $p < 0,05$ . O PDL obteve valor médio de 956,0 NCF (352,0 - DP) e o PTN alcançou 391,0 NCF (36,7 - DP) com significância estatística entre os grupos ( $p < 0,001$ ). Não houve diferença estatística no comprimento do fragmento ( $p > 0,05$ ). A análise SEM mostrou características compatíveis com fratura dúctil. Os arquivos ProDesign LOGIC apresentaram NCF mais alto que o Protaper Next X2. Não houve diferença entre os grupos considerando o comprimento do fragmento rompido.

**Palavras-chave:** Endodontia; Instrumentos odontológicos; Rotação.

### Resumen

El objetivo de este estudio fue evaluar la resistencia a la fatiga cíclica de limas novedosas de NiTi (sometidas a tratamiento térmico) utilizando el modelo in vitro (canal artificial). Doce instrumentos ProDesign Logic - PDL 25/.06 (Easy Equipamentos Odontológicos, Belo Horizonte, Brasil) y 12 instrumentos Protaper Next X2 - PTN (tip 25) fueron incluidos en esta investigación. El ensayo de fatiga cíclica estática se realizó con un bloque ranurado de acero inoxidable simulando un canal de 1,5 mm de diámetro, un ángulo de curvatura de  $60^\circ$  y un radio de 5 mm. Las limas se colocaron dentro del canal artificial y se rotaron hasta que se produjo la fractura. Utilizando el tiempo hasta la fractura (segundos) y el número de rotaciones por minuto (RPM), se calculó el número de ciclos hasta la fractura (NCF) y se registró la longitud de los fragmentos rotos. Tres muestras de cada grupo fueron analizadas en Microscopía Electrónica de Barrido (SEM) para caracterizar la ruptura del metal. Los datos fueron analizados mediante la prueba no paramétrica de Mann-Whitney y el nivel de significancia considerado fue  $p < 0,05$ . PDL obtuvo un valor medio de 956,0 NCF (352,0 - DE) y PTN alcanzó 391,0 NCF (36,7 - DE) con significación estadística entre los grupos ( $p < 0,001$ ). No hubo diferencia estadística en la longitud del fragmento ( $p > 0,05$ ). El análisis SEM mostró características compatibles con fractura dúctil. Los archivos ProDesign LOGIC mostraron un NCF más alto que Protaper Next X2. No hubo diferencia entre los grupos considerando la longitud del fragmento roto.

**Palabras clave:** Endodoncia; Instrumentos dentales; Rotación.

## 1. Introduction

During endodontic treatment in curved canals, the root anatomy determines the greater or lesser curvature of the instrument (Kim et al., 2009). The superelastic nature of NiTi, from which the new instruments are made, is attributed to the reversible transformation of austenite into martensite. The austenite becomes martensite during loading and reverts to austenite with the removal of tension. Loading and unloading cycles accumulate at each complete revolution of the instrument, progressively altering the mechanical behavior of the NiTi alloy and finally leading to a rupture by cyclic fatigue fracture (Kuhn & Jordan, 2002; Lee et al., 2011). Therefore, the number of cycles to fracture (NCF) has been a commonly cited parameter to evaluate and compare the lifespan of files considering metal fatigue.

In an attempt to improve the performance of the instruments to cyclic fatigue, new methods of fabrication or pretreatment of the alloy have been used to increase file resistance to successive loading cycles. The thermal treatment of NiTi instruments is based on the relationship between the phases: austenitic, martensitic and R-phase. During cooling, austenite is transformed into R phase, and then martensite (direct transformation). Heating changes the martensite into R phase and then into austenite (reverse transformation). The mechanical properties and temperature of the phase changes of the alloy depend directly on the thermal processing. The knowledge and appropriate control of these treatment protocols is essential for the use of this metal alloy in its various applications (Kuhn & Jordan, 2002; Viana et al., 2010). In this sense, in 2007 the second generation of NiTi files was introduced, which was called M-Wire (Memory wire) (Dentsply Tulsa Dental Specialties, Tulsa, OK). These instruments were subjected to heat treatment and implicated in presenting greater resistance to cyclic fatigue and flexibility than the traditional NiTi alloy (Pereira et al., 2012). In 2009, a new heat treatment process created the CM-Wire (Controlled Memory Wire) alloy (Typhoon CM; Clinican's Choice Dental Products, New Milford, CT). These new instruments were also implicated in exhibiting greater cyclic fatigue resistance than instruments made from the conventional NiTi superelastic alloy (Shen et al., 2011).

The ProTaper Next files (Dentsply Maillefer, Ballaigues, Switzerland) are newly introduced M-Wire instruments, which have a variable taper and a decentralized rectangular cross-section (Uygun et al., 2016). New types of heat treatments and new instruments have been continuously produced. The ProDesign Logic instruments (Easy Dental Equipment, Belo Horizonte, Brazil) were created from these new methods. These are CM-Wire alloy instruments with a modified S-Shaped cross-sectional design, which can operate in rotation or in reciprocal motion, according to the manufacturer.

To date, few studies have been performed in the literature comparing the cyclic fatigue resistance of the new, recently created instruments. Thus, the present study has the objective of comparatively evaluating the cyclic fatigue resistance of these NiTi new instruments through an in vitro model (artificial canal).

## 2. Methodology

The methodology used in this research was based on the study of Gündoğar and Özyürek (Gündoğar & Özyürek, 2017). Briefly: for group A, 12 ProDesign Logic (PDL) instruments (tip 25 taper .06) and for group B, 12 Protaper Next (PTN) X2 instruments (tip 25, taper .06 at 5 mm from the tip) were selected. For the static test of cyclic fatigue strength performed at room temperature (25°C), an artificial stainless steel canal was used. The straight portion of the conduit (cervical segment) was 10 mm long. The canal was 1.5 mm wide, has 2.0 mm of groove depth, 19.0 mm total length, a 60° angle of curvature and a curvature radius of 5 mm (fig.1). In both groups, the conduit was lubricated with a synthetic lubricant (WD-40 Company, Milton Keynes, England) to minimize friction between the canal and the files, ensuring free rotation within the artificial canal.

**Figure 1.** Artificial canal: Straight portion (cervical segment) with 10mm, 1.5mm wide, 2.0 mm of groove depth, 19.0 mm of total length, 60° angle of curvature and 5 mm radius.



Source: Authors.

Each instrument was inserted 19 mm into the artificial canal. Rotation speeds (RPM) and torques were set according to the manufacturer's determinations: 950 RPM and 4N/cm<sup>2</sup> for group A (PDL), 300 RPM and 2N/cm<sup>2</sup> for group B (PTN). The instruments were rotated in the canals until the fracture occurred. The time until fracture was registered using 3 digital stopwatches. Using the mean of the obtained times, the number of cycles until fracture (NCFs) were calculated according to

the following formula:  $NCFs = RPM \times \text{time until fracture (seconds)} / 60$ . The lengths of the fragments were measured using a magnifying glass. Six instruments (n = 3 from each group) were examined with a scanning electron microscope (SEM) (TM 3000 - Tabletop Microscope; HITACHI, Tokyo, Japan) in order to confirm that the instruments were actually ruptured by cyclic fatigue.

The data were evaluated using the Mann-Whitney Non-Parametric test (for samples without normal distribution). The level of significance was set at  $p < 0.05$ . Minitab 19 statistical software was used to accomplish the analysis of the data.

### 3. Results

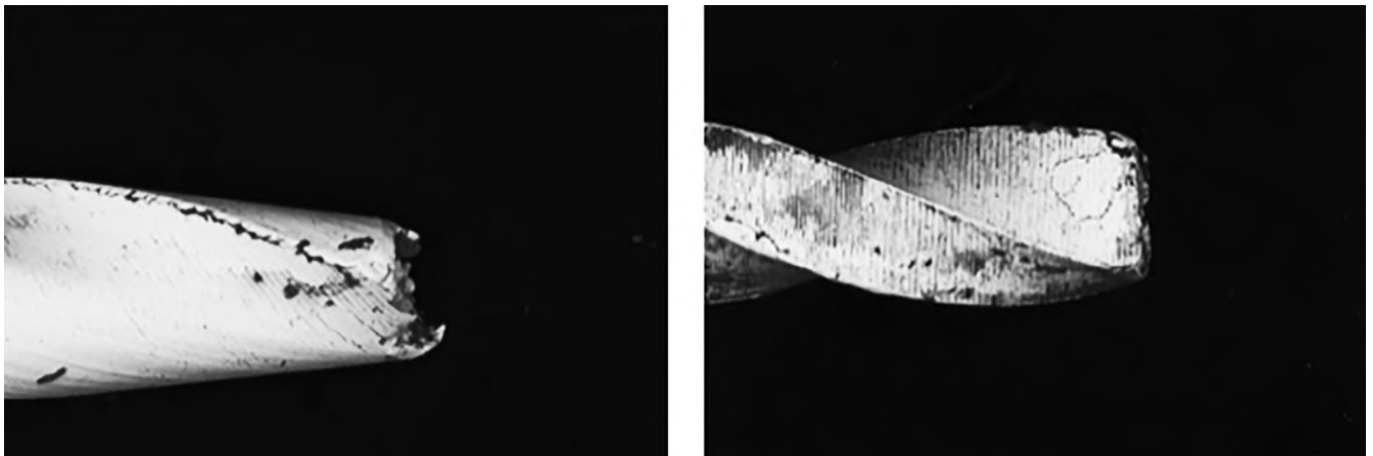
Table 1 shows the means and standard deviation of the NCF (number of cycles to fracture) and length of the broken fragment of groups A and B. The PDL group: NCF = 956,0 (352,0) - presented higher resistance to flexural fatigue than the PTN group: NCF = 391,0 (36,7). There was no statistically significant difference between the groups in relation to the length of the ruptured fragment ( $p > 0.05$ ).

**Table 1.** Mean Values (standard deviation) for Number of Cycles to Fracture (NCF) and Fragment Length.

GROUP	INSTRUMENT	NCF (SD)	FRAGMENT LENGTH mm (SD)
A (PDL)	LOGIC 25.06	956,0 (352,0)	4,4 (0,4)
B (PTN)	PROTAPER NEXT X2	391,0 (36,7) **	4,5 (0,4) *

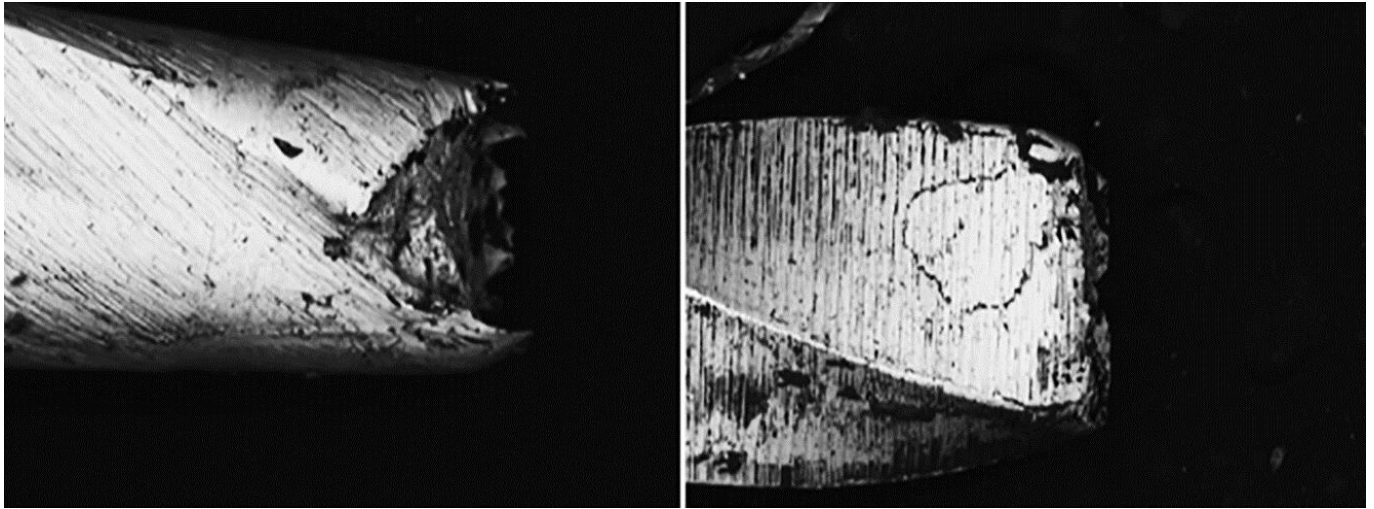
\*\*statistically significant differences ( $p < 0.001$ ) / \* no statistically significant differences  $p > 0.05$ . Source: Authors

**Figure 2.** Scanning electron microscopy images of groups A (PDL) left and B (PTN) right (x100) taken laterally, showed absence of plastic deformation in the region of the rupture, ruling out the possibility of torsional fracture.



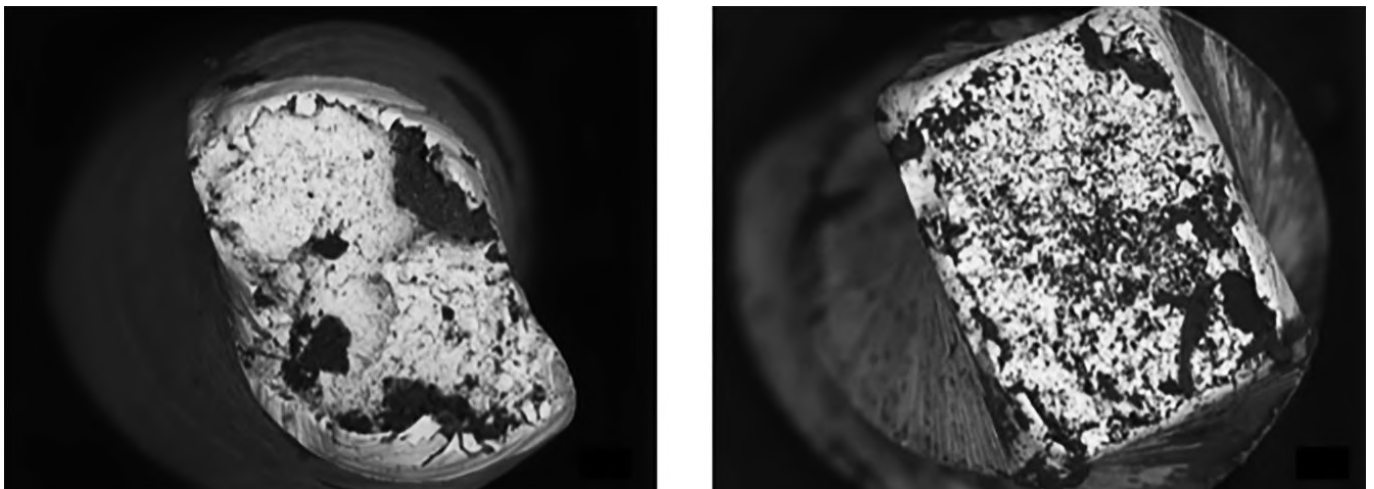
Source: Authors.

**Figure 3.** Scanning electron microscopy images of groups A (PDL) left and B (PTN) right (x200) taken laterally showing details of the region of rupture.



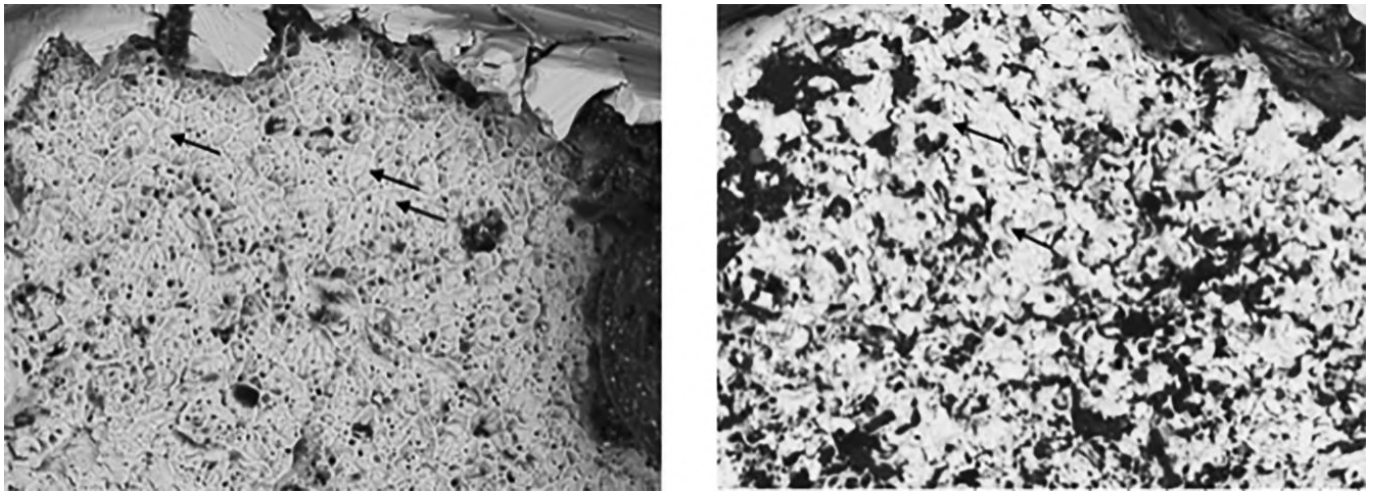
Source: Authors.

**Figure 4.** In the images of the S-shaped cross-sections of group A (PDL) left and the rectangular cross sections of group B (PTN) right.



Source: Authors.

**Figure 5.** In the larger view images (x1000), the presence of microcavities (dimples - arrows) indicating ductile fracture are observed. The characteristics of the NiTi alloy are compatible with this type of fracture, generating crack development at the edge of the microcavities, promoting a continuous and gradual rupture of the material.



Source: Authors.

It is very important to note that the aspect of the rupture reflect exactly the pattern presented by continuous degradation of the alloy, confirming that the rupture occurred due to fatigue and not torsion.

#### 4. Discussion

The rupture of instruments within the root canals represents one of the main types of accidents observed during endodontic treatment, becoming a particularly evident situation more than 20 years ago at the beginning of the instrumentation through rotating NiTi files. At that time, fracture by torsion fatigue, when the tip of the instrument was attached to the walls of the channels while the remainder of the instrument remained spinning (Kitchens et al., 2007), was the most common type of fracture (Sattapan et al., 2000). However, the implementation of torque control equipment, the development of more accurate and safer techniques, as well as greater knowledge regarding the limitations of the instruments, reduced the possibilities of torsional fatigue and placed fracture by cyclic fatigue at the center of the studies. Currently, most studies consider that fracture by cyclic fatigue is the main cause of separation of NiTi instruments (Shen et al., 2009; Bhagabati et al., 2012). Thus, the evaluation of the number of cycles to fracture (NCF), which is the central objective of the present study, to estimate the wear of the alloy and the useful life of the instruments becomes an important parameter of comparison between new instruments. Shen et al. (Shen et al., 2009) analyzed 1682 instruments collected after normal clinical use in a cohort study for a period of 16 months. Although there is no consensus regarding the maximum number of uses to which the instruments can be submitted in the literature, this work shows that only 0.5% of new files fractured in their first clinical use.

The methodology used in this study is well established in the scientific community, and has been replicated in numerous studies in different research centers around the world. Thus, the morphometric characteristics of the artificial canal (60° of curvature, 1,5 mm internal diameter and 5mm radius of curvature) that conformed to the world-wide specifications can allow the obtained results to be compared to other research with the same parameters. Although the use of extracted teeth could better simulate real clinical conditions, the differences in anatomical features from tooth to tooth would be virtually impossible to standardize. In this case we could not conclude that the results obtained were due only to the experimental parameters.

Among the factors that influence the number of cycles to fracture, those most directly related, and therefore most cited

by the authors, are anatomical (angle and radius of curvature) (Haikel et al., 1999; Pirani et al., 2011; Gündoğar & Özyürek, 2017) and those inherent to the instrument, such as design, kinematics, cross sectional area, taper, method of manufacture, constitution and treatment of the alloy (Shen et al., 2009; De-Deus et al., 2010; Cheung et al., 2011; Wealleans et al., 2011; Bhagabati et al., 2012; Özyürek et al., 2017; Ruiz-Sánchez et al., 2020; Hua et al., 2021). In the present study, although some of these factors are the same among the evaluated instruments, others are different and may influence the observed results. The anatomical factors (angle and radius of curvature), kinematics, taper at 5mm from the tip, constitution and manufacturing method are items in which the evaluated instruments are equivalent. However, the factors of design, cross sectional area and treatment of the metallic alloy in the area are great differences that can influence the obtained results, exerting greater or smaller influences.

In the present study, we compared files with different cross-sectional designs. Özyürek and colleagues (Özyürek et al., 2017) also compared cyclic fatigue resistance of s-shaped cross sectional files with quadrangular cross sectional files. Although the s-shaped files obtained better results, like our investigation, it is important to mention that these files also presented lower cross-sectional areas in the region of instrument rupture. It is generally accepted that files with lower tapers, therefore less metallic mass, are more resistant to flexural fatigue tests. It would be reasonable to assume that the s-shaped cross-sectional area of Logic files is lower than the rectangular cross-sectional area of Protaper Next; however, we have no data to consider this feature as principally responsible for the results. As a limitation of our work, we didn't measure the cross-sectional area of the files at the rupture site, and perhaps this data could help to better discuss the results we achieved. In another study, when instruments with similar cross-section designs were compared for cyclic fatigue resistance, the authors concluded that better results were influenced by the surface treatment (Kwak et al., 2017).

In the present study, we compared files with different thermal treatments of the NiTi alloy, the CM-wire alloy of the Logic files and M-wire alloy of the Protaper Next files. Some studies highlight the greater influence of the "alloy treatment" factor in relation to the other features of the evaluated instruments. Shen et al. (Shen et al., 2011) promoted the metallurgical characterization of the treated NiTi alloy CM-Wire. The authors concluded that CM-Wire alloy instruments have a final austenitic transformation temperature above body temperature, so they present a high percentage of martensite at room temperature. In a complementary study, Shen and colleagues (Shen et al., 2011) examined the fatigue behavior of the new CM-Wire alloy compared to the traditional alloy. The researchers found that the instruments constituted by the CM-Wire alloy presented significantly more cycles to fracture than traditional instruments. In addition, it was observed that CM-Wire instruments generate a greater number of crack origin points than conventional alloy instruments. Although LOGIC (CM-Wire) instruments have few studies in the literature, some studies comparing NCF between M-Wire and CM-Wire instruments obtained results similar to the present study. Capar and colleagues (Capar et al., 2015) comparatively evaluated the flexural fatigue resistance of ProTaper Next X2 (M-Wire), OneShape (conventional NiTi), Revo-S Shaping Universal and HyFlex 25 / 0.6 (CM wire) instruments. The authors concluded that CM-Wire instruments presented greater resistance to fatigue than all other groups. In another study, Capar et al. (Capar et al., 2015) evaluated pathfinder instruments produced by traditional NiTi, M-Wire and CM-Wire. The researchers concluded that CM-Wire instruments were the most resistant to flexural fatigue compared to the instruments of all other groups. Recent studies have evaluated other CM-Wire instruments in relation to fatigue resistance, obtaining similar results. Alcalde et al. (Alcalde et al., 2017) and Silva et al. (Silva et al., 2016) also concluded that the EASY ProDesignR (CM-Wire) instrument obtained the best results regarding fatigue resistance compared to the other instruments evaluated. Menezes et al. (de Menezes et al., 2017) compared the NCF in artificial canals, between WaveOne Gold instruments (Dentsply Maillefer, Ballaigues, Switzerland) and ProDesign LOGIC 25.06. These authors concluded that ProDesign LOGIC presented high NCF than WaveOne Gold.

It is important to emphasize that the results obtained in *in vitro* studies should not be directly transported to *in vivo*

working conditions. In real clinical situations, the instruments face other challenges such as calcifications, complex anatomies, atresia conducts, among others. Moreover, the motion of the instruments differs considerably from the movement used in static studies of flexural fatigue (the present work), when they remain rotating at the same depth of penetration. Clinically, each instrument moves continuously along the axial axis of the canals. In this respect, the dynamic studies of flexural fatigue that perform this axial movement can approximate the results obtained from the clinical situations, not presenting the limitation presented by the present study. However, higher NCF values, even in static tests, indicate greater resistance of one instrument over another. This may imply longer life and a greater number of uses, provided that the other clinical factors involved are taken into account. Therefore, considering the importance of the number of uses of each instrument in the clinical protocols, new studies comparing the NCF, using dynamic movement should be proceeded.

Considering the results obtained in relation to the size of the fragment, as observed in the research of Gündoğar et al. (Gündoğar et al., 2017), no significant difference was observed between the groups. This result confirms that all instruments tested were positioned in the same region of maximum stress as the artificial conduit curvature.

## 5. Conclusion

According to the conditions of the present study, Prodesign LOGIC instruments (25.06) obtained a greater number of cycles until fracture (NCF) than Protaper Next X2 instruments (statistically significant difference ( $p < 0.001$ ). There was no statistically significant difference between the groups in relation to the length of the ruptured fragment ( $p > 0.05$ ).

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