UNIVERSIDADE DE SÃO PAULO
FACULDADE DE ODONTOLOGIA DE BAURU

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Analysis of mechanical properties engine-driven NiTi instruments with different thermally treated

Análise das propriedades mecânicas de instrumentos rotatórios de NiTi com diferentes tratamentos térmicos

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Orientador: Prof. Dr. Marco Antonio Hungaro Duarte

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FOLHA DE APROVAÇÃO

DEDICATÓRIA

A Deus,

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RESUMO

Obejtivo: Artigo 1- avaliar o impacto da temperatura corporal a resistencia a fadiga cíclica de diferentes Ligas de NiTi usadas na fabricação dos instrumentos: Reciproc Blue (Vdw Munich), X1 Blue (MK Life, Porto Alegre), Waveone Gold (Dentsply Ballaigues). Artigo 2- avaliar a resistência à fadiga cíclica e torcional de instrumentos rotatórios de níquel-titânio com secções transversais semelhantes e confeccionados com diferentes tipos de tratamentos térmicos: Hyflex CM (HCM 25/.06), Vortex Blue (VB 25/.06), Sequence Rotary File (SRF 25/.06) e EdgeSequel (EDF 25/.06) (n=20). **Metodologia:** Artigo 1- 60 instrumentos de Reciproc Blue, X1 Blue e Waveone Gold foram usados (n=20). Teste de fadiga cíclica foram realizados em temperatura ambiente (20º C + - 1) e a temperatura corporal (37º +-1). Os instrumentos foram acionados no movimento reciprocante ate ocorrer a fratura em um canal artificial de aço inox com curvatura num ângulo de 60º e 5mm Raio. O Tempo de frartura foi registrado TTF foi registrado. Tambem o Numero de Ciclos para fratura NCF foi calculado. Artigo 2- O teste de fadiga cíclica avaliou o tempo TTF e o número de ciclos para a fratura (NCF) dos instrumentos em canal artificial com 60° de curvatura e 5mm de raio (n=10). O teste de torção avaliou o torque máximo e a deflexão angular para a fratura dos 3mm da ponta dos instrumentos (n=10). Após o teste de torção e de fadiga cíclica, os instrumentos foram avaliados em microscopia eletrônica de varredura (MEV) (n=10). Os dados foram analisados empregando-se os testes ANOVA e de Tukey, sendo selecionado um nível de significância de5%. **Resultados:** Artigo 1- O teste da fadiga cíclica a 20º C mostrou que Reciproc Blue 25/08 e X1 Blue 25/06 apresenta significantemente maior TTF a NCF que Waveone Gold 25/07.Em 37º C, todos os grupos apresentaram significante redução do TTF e NCF. Reciproc Blue 25/08 apresentou significantemente amaior TTF que Waveone Gold 25.07. Referente ao NCF não houve diferença significante entre os grupos. Waveone Gold 25.07 apresentou menor porcentagem de redução no teste de fadiga cíclica. Artigo 2- HCM apresentou o maior tempoe NCF entre todos os instrumentos avaliados (p0,05) e menor NCF (p0,05). **Conclusao**: Artigo 1- A temperatua corporal provoca uma significativa redução na resistência a fadiga cíclica de todos os intrumentos reciprocantes testados. Reciproc Blue e X1 Blue apresentaram similar resultados em ambas temperaturas testadas. Waveone Gold apresentou menor porcentagem de redução a resistência a fadiga na temperatura corporal. Artigo 2- O HCM apresentou maior resistência à fadiga cíclica e deflexão angular. Entretanto, o VB apresentou maior resistência torcional para a fratura.

Palavras-chave: Instrumentos odontológicos. Endodontia. Níquel. Titânio.

ABSTRACT

Objective: article 1- The aim of this study was to evaluate the impact of body temperature on the cyclic fatigue resistance of different NiTi alloys used for the manufacturing of Reciproc Blue R25 (RB 25.08; VDW, Munich, Germany), X1 Blue File 25 (X1 25.06; MK Life Medical and Dental Products, Porto Alegre, Brazil) and WaveOne Gold Primary (WOG 25.07; Dentsply Maillefer, Ballaigues, Switzerland). Article 2- evaluate the cyclic and torsional fatigue resistance of Nickel-Titanium rotary instruments with similar cross sectional design and manufactured by different thermal treatments. **Methodology** Article 1- Sixty instruments of the RB 25.08, X1 25.06 and WOG 25.07 systems were used (n = 20). Cyclic fatigue tests were performed at room temperature (20° ± 1 °C) and at body temperature (37° ±1 °C). The instruments were reciprocated until fracture occurred in a stainless steel artificial canal with a 60° angle and a 5-mm radius of curvature. The time to fracture (TTF) was recorded. Also, the number of cycles to fracture (NCF) was calculated. Data were analysed using one-way ANOVA and Tukey's tests for inter-group comparison at both temperatures and for the reduction of cyclic fatigue at body temperature. For intra-group comparison at the different temperatures, the unpaired t test was used. Article 2- eighty instruments of Hyflex CM (HCM; #25/.06) Vortex Blue (VB; #25/.06), Sequence Rotary File (SRF; #25/.06) and EdgeSequel (EDF #25/.06) were used (n=20). Cyclic fatigue test evaluated the time and number of cycles to failure (NCF) in a stainless steel artificial canal with 60° and 5-mm radiusof curvature (n=10). The torsional test (ISO 3630-1) evaluated the maximum torque and distortion angle to failure in the 3 mm from the tip (n=10). The topographic features of fractured surface of instruments were assessed using scanning electron microscopy (SEM). Data were analyzed using one-way ANOVA and Tukey tests, and the level of significance was set at 5%.**Results:** Article 1- The cyclic fatigue test at 20 °C showed that RB 25.08 and X1 25.06 presented significantly higher TTF and NCF than WOG 25.07 ($P < 0.05$). At 37 °C, all groups presented significant reduction of TTF and NCF ($P < 0.05$). RB 25.08 presented significant higher TTF than WOG 25.07 ($P < 0.05$). Regarding the NCF, there was no significant difference among the groups ($P > 0.05$). The WOG 25.07 presented the lowest percentage reduction of cyclic fatigue ($P < 0.05$). Article2- The HCM presentedthe longest time and highest NCF to cyclic fatigue compared with all the groups ($P<0.05$). The SRF presented similar time as, ($P<0.05$) and lower NCF ($P<0.05$) to fatigue than VB. Relative to the torsional test, HCM presented the lowest torque load and the highest distortion angleof all

the groups(P<0.05). The SRF and EDF presented similar torque load (P>0.05). There was no difference among VB, SRF and EDF regarding the distortion angle (P>0.05). The SEM analysis showed typical features of cyclic and torsional fatigue for all instruments tested. **Conclusion**: Article 1- The body temperature treatment caused a marked reduction of the cyclic fatigue resistance for all reciprocating instruments tested. The RB 25.08 and X1 25.06 systems presented similar results at both temperatures tested. However, WOG 25.07 presentedthe lowest percentage reduction in fatigue resistance at body temperature. Clinical relevance Cyclic fatigue resistance of NiTi reciprocating instruments has been evaluated at room temperature. However, the fatigue resistance significantly decreases upon exposure to body temperature, which could affect the mechanical behaviour of the NiTi instruments during root canal preparation. Article 2- The HCM presented the highest cyclic fatigue resistance and angular rotation to failure. However, the VB showed higher torsional strength to failure.

Keywords: Cyclic Fatigue, torsional resistance, rotary systems, thermal treatment, nickel-titanium.

SUMÁRIO

1 INTRODUCTION

1 INTRODUCTION

The Nickel-Titanium alloy (NiTi) was introduced in endodontics in 1988 by Walia *et al.* The authors demonstrated that prototype of NiTi hand files were more flexible andpresented greater deformation capacity by torsion than stainless steel hand files, which could be advantage during root canal preparation of curved canals (GAVINI *et al.*, 2018). Since then, several studies were published reporting that NiTi instruments provide centering root canal shaping and safety endodontic root canal preparation in comparison with stainless steel endodontic instruments (PETERS; SCHONENBERGER; LAIB, 2001; PETTEITE; DELANO; TROPE, 2001; PETERS, 2004; CHEUNG; LIU, 2009)

The NiTi alloy (55% Nickel; 45% Ti) present low modulus of elasticity, high flexibility, shape memory effect and high recoverable strains (OUNSI *et al.*, 2017; GAVINI *etal.*, 2018). The shape memory is defined as the ability of the NiTi to deform and recover to the original shape. The superelasticity is the ability of the NiTi alloy to suffer large deformation previously to fracture. These aforementioned features exist due to two equiatomic phases, the Austenite (B2) and Martensite (B19) (THOMPSON, 2000). In the Austenite phase the atoms are in a cubic crystalline form and it is responsible to shape memory effect, which can be induced by tension or heating. On the other hand, when the NiTiis undergone by tension or cooling, the atoms are rearranged into a monoclinic crystalline structure with provides superelasticity, called Martensite Phase (THOMPSON, 2000). It is important to highlight that the austenite-martensite transformation is a reversible process, differently from stainless steel alloy (THOMPSON, 2000; BAUMANN, 2004)

The proposal of Walia *et al.* to manufacture endodontic instruments using NiTi alloy allowed that curved canals could be also mechanically prepared using a continuous rotary motion instead of only hand files (GAVINI *et al.*, 2018; HAAPASSALO; SHEN, 2013). The mechanical properties of NiTi alloy allowed safety during root canal preparation especially in curved canals, reducing the risk of instruments separation caused by cyclic or torsional fatigue (SATTAPAN *et al.*, 2000; PLOTINO *et al.*, 2014). The cyclic fatigue occurs by repeated compressive and tensile stresses at the maximum point of flexion when the instrument rotates in a curved canal (SATTAPAN *et al.*, 2000). Torsional failure occurs when the tip of the instrument is locked in the canal whilst the shank continues to rotate (SATTAPAN *et al.*, 2000). This can happen in straight or curved canals, especially in the preparation of narrow and constricted canals when the file is susceptible to high torsional loads (SATTAPAN *et al.*, 2000). Torsional resistance is characterized by a maximum torsional load and angle of rotation. This property reveals the ability of the file to twist before fracture (WYCOFF; BERZINS, 2012).

During the last decades, several studies evalauted the cyclic and torsional resistance to fatigue of the NiTi rotary instruments, which showed that the instruments design (cross- section desing, taper, diameter of core, tip diameter) can affect the metal mass volume of the instruments, modifying their flexibility and clinical performance (TOPÇUOGLU *et al.*, 2016; WYCOFF; BERZINS, 2012; USLU *et al.*, 2017; ALCALDE *et al.*, 2017, 2018, 2020). Therefore, the manufacturers began to develop other methods to improve the resistance to fatigue.

The most of brands of NiTi engine-driven endodontics instruments are mainly manufactured by machining process, which could cause micro defects of the instrumentsurfaces (LOPES *et al.*, 2010; GAVINI *et al.*, 2018). The micro defects on the surfaces can lead to a crack on the instruments, increasing the risk of instrument separation during root canal preparation (LOPES *et al.*, 2010; HAAPASSALO; SHEN, 2013). Thus, the manufacturer developed a new method to improve reduce these micro defects on the instruments surface after machining process, called as electropolishing (LOPES *et al.*, 2010; HAAPASSALO; SHEN, 2013). The BioRaCe was the first rotary system manufactured with this technology and several authors demonstrated that has a significant reduction of the imperfections on the instrument’s surface and, also, improved the cyclic fatigue resistance (RAPISARDA *et al.*, 2000; SHEN *et al.*, 2013; LOPES *et al.*, 2010; GUTTMAN; GAO, 2012).

After the introduction of electropolishing method, the manufacturers developed others technologies in order to optimize the mechanical properties of NiTi instruments, modifyingthe microstructure of atoms the NiTi alloy (HAAPSALO; SHEN, 2013; SHEN *et al.*, 2013). The thermal treatment of the NiTi alloy is a technology that assists to control the austenite-martensite transformation and induce a better arrangement of the crystal structure using a controlled heating-cooling process (SHEN *et al.*, 2013; ZUPANC *et al.*, 2018). This treatment of the NiTi provides greater flexibility and deformation capacity under high mechanical stress in comparison with conventional NiTi alloy (SHEN *et al.*, 2013; ZUPANC *et al.*, 2018). The thermal treatments increase the percentage of martensite phase (which is known to be more flexible than austenitic NiTi) or R-Phase (an intermediate phase between the austenite and martensite phase) in ambient temperature, improving the resistance to fatigue (SHEN *et al.*, 2013; GAVINI *et al.*, 2018; ZUPANC *et al.*, 2018). Also, some type of thermal treatment allow that the spiral flutes of the instruments can be manufacture by twisting process insteadof machining method because of the R-phase features (LOPES *et al.*, 2013; ZUPANC *et al.* 2018).

Another method of manufacturing process was introduced in 2015 by Coltene (Coltène Whaledent, Switzerland) in a new rotary system, Hyflex EDM (Coltène Whaledent, Switzerland). This rotary system is manufactured via an electrical discharge machining (EDM) process that provides a harden the surface of the NiTi file, resulting in an improved fracture resistance and superior cutting efficiency (ZUPANC *et al.*, 2018). EDM process is a well-known noncontact machining procedure that allows precise material removal via pulsed electrical discharge (BOJORQUEZ *et al.* 2002, DANESHMAND *et al.* 2013). Embedded in a dielectric liquid, the machining tool is moved toward the workpiece until the gap is small enough so that the applied voltage is able to ionize the dielectric liquid (BOJORQUEZ *et al.* 2002). The resulting spark vaporizes small particles from the workpiece, which resolidify in the dielectric liquid and are subsequently flushed away (BOJORQUEZ *et al.* 2002), EDMdoes not require direct contact with the workpiece, which eliminates the chance of mechanicalstress as in the traditional grinding process (SINGH *et al.* 2004). In addition, this process can modify a phase composition in the crystalline structure, improving the flexibility.

Currently, there are nearly of seven types of thermal treatment used in NiTi instruments: Martensite Wire (M-Wire/ 2007), R Phase NiTi (2008), Controlled Memory Wire (CM-Wire/ 2010), Blue Wire (2013), Gold Wire (2014), EDM (2015) and Max-Wire (2015) (SHEN *et al.*, 2013; GAVINI *et al.*, 2018; ZUPANC *et al.*, 2018). The main differenceamong these different NiTi treatments is the percentage of martensite phase in room temperature, consequently, different cyclic and torsional fatigue resistance (ZUPANC *et al.*, 2018).

Although the development of several rotary instruments with different design and the thermal treatments, instrument separation continues to be an undesirable occurrence during endodontic procedures (GAMBARINI *et al.*, 2015; ALCALDE *et al.*, 2018). In 2008, Yared proposed a new kinematics to be used during canal preparation, called reciprocating motion

(DE-DEUS *et al.*, 2010; KIM *et al.*, 2012). This kinematicas involves rotation in counterclockwise (CCW) and clockwise (CW) directions with 120° of difference between the two movements and completes 360° in 3 cycles (KIM *et al.*, 2012). This kinematics have some advantages in comparison with rotary motion, such as: low screw-in effect, less cyclic fatigue stress and less torsional stress, reducing the risk of instrument separation during canal preparation of curved and constricted canals (VALERA-PATIÑO *et al.*, 2010; KIM *et al.*, 2012; GAVINI *et al.*, 2018).

The advances on the metallurgy and kinematics of the engine-driven NiTi instruments improved the safety during root canal preparation of curved and constricted canals (SHEN *et al.*, 2013; GAVINI *et al.*, 2018). In addition, the most recent NiTi instruments have a significant improvement on the cyclic and torsional fatigue resistance in comparison with the firsts systems developed in the beginning of 1990 (GAVINI *et al.*, 2018).

The cyclic and torsional fatigue test of the engine-driven rotary instruments aimed to simulate a mechanical stress caused during root canal preparation curved or constricted canal, assisting in to understand how would be the mechanical behavior the of the instruments in different root anatomies (ALCALDE *et al.*, 2017, 2018). The most part of the mechanical test are performed at room temperature, however, after a better comprehension of the austenite-martensite transformation and the thermal treatments, some studies have demonstrated that room temperature can affect the mechanical properties of the instruments (ALCALDE *et al.*, 2018; STAFFOLI *et al.*, 2019; ARIAS *et al.*, 2019).

Depending of the type of thermal treatment performed during manufacturing processof the NiTi instruments the room temperature can induce a crystalline alteration (modifying the crystalline phase). The transition temperature depends of the initial (Start – S – the phase begins to appear) and final (Final – F – phase transformation completed) limits induced by thermal treatment or type of the alloy. A modified phase composition due to changed transformation temperature is the main difference between the different NiTi engine-drive instruments. Therefore, these modifications can lead to more flexible endodontic instruments with an advanced resistance to fracture (ZUPANC *et al.*, 2018; STAFFOLI *et al.*, 2019).

The Austenite Final temperature (Af) of NiTi induced by the thermal treatments process of the instruments should be higher than root canal temperature (35-36^{0}C), ideally, to avoid the less flexible crystalline phase, the austenite phase, and ensure more flexible and resistant instruments clinically. The Af temperature of the conventional NiTi (18-31^{0}C), R-Phase (17^{0}C), M-Wire (50^{0}C), Blue (38^{0}C), Controlled Memory Wire (55^{0}C), Gold (50^{0}C), Max-Wire (35^{0}C) and Electrical Discharge machining (60^{0}C) differed among than according to studies of differential Scanning Calorimetry (DSC). Therefore, these types of NiTi present different crystalline phase when exposed on root canal temperature, consequently, different mechanical properties (ZUPANC *et al.*, 2018; STAFFOLI *et al.*, 2019, ARIAS *et al.*, 2019).

The evaluation of the cyclic and torsional resistance of NiTi engine-drive instruments need to considered several factors, such as instruments design (cross-section, taper, diameter of core and tip diameter) and the different thermal treatments of the NiTi, which has a key role effect on their mechanical properties and clinical performance. For this reason, the comparison of instruments with different features is a complex process (PEDULLA *et al.*, 2016)

Currently, there are several rotary and reciprocating NiTi instruments with different design and thermal treatments for root canal preparation. Thus, it is important for the clinician to know the cyclic and torsional properties of the instruments to provide a safe endodontic procedure.

The aim of this study was to evaluate the cyclic and torsional fatigue resistance of reciprocating and rotary instruments manufactured by different NiTi alloy. For this purpose, the study was performed in tree steps:

- **1st article:** to evaluate the effect of temperature on the cyclic fatigue resistance of thermally treated reciprocating instruments.

- **2nd article:** to evaluate the cyclic and torsional fatigue of four NiTi rotary instruments with similar cross-section and different thermal treatments.

2 ARTICLES

2 ARTICLES

2.1 ARTICLE 1 – EFFECT OF TEMPERATURE ON THE CYCLIC FATIGUE RESISTANCE OF THERMALLY TREATED RECIPROCATING INSTRUMENTS

The first article presented in this thesis was published in the *Clinical Oral Investigation*.

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ORIGINAL ARTICLE

Effect of temperature on the cyclic fatigue resistance of thermally treated reciprocating instruments

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Abstract

Objectives The aim of this study was to evaluate the impact of body temperature on the cyclic fatigue resistance of different NiTi alloys used for the manufacturing of Reciproc Blue R25 (RB 25.08; VDW, Munich, Germany), X1 Blue File 25 (X1 25.06; MK Life Medical and Dental Products, Porto Alegre, Brazil) and WaveOne Gold Primary (WOG 25.07; Dentsply Maillefer, Ballaigues, Switzerland).

Materials and methods Sixty instruments of the RB 25.08, X1 25.06 and WOG 25.07 systems were used ($n = 20$). Cyclic fatigue tests were performed at room temperature (20° ± 1 °C) and at body temperature (37° ± 1 °C). The instruments were reciprocated until fracture occurred in an artificial stainless steel canal with a 60° angle and a 5-mm radius of curvature. The time to fracture (TTF) was recorded. Also, the number of cycles to fracture (NCF) was calculated. Data were analysed using one-way ANOVA and Tukey's tests for inter-group comparison at both temperatures and for the reduction of cyclic fatigue at body temperature. For intra-group comparison at the different temperatures, the unpaired *t* test was used.

Results The cyclic fatigue test at 20 °C showed that RB 25.08 and X1 25.06 presented significantly higher TTF and NCF than WOG 25.07 ($P < 0.05$). At 37 °C, all groups presented significant reduction of TTF and NCF ($P < 0.05$). RB 25.08 presented significant higher TTF than WOG 25.07 ($P < 0.05$). Regarding the NCF, there was no significant difference among the groups ($P > 0.05$). The WOG 25.07 presented the lowest percentage reduction of cyclic fatigue ($P < 0.05$).

Conclusion The body temperature treatment caused a marked reduction of the cyclic fatigue resistance for all reciprocating instruments tested. The RB 25.08 and X1 25.06 systems presented similar results at both temperatures tested. However, WOG 25.07 presented the lowest percentage reduction in fatigue resistance at body temperature.

Clinical relevance The cyclic fatigue resistance of NiTi reciprocating instruments has been evaluated at room temperature. However, the fatigue resistance significantly decreases upon exposure to body temperature, which could affect the mechanical behaviour of the NiTi instruments during root canal preparation.

Keywords Cyclic fatigue · Nickel titanium · Reciprocating systems · Body temperature

Introduction

Engine-driven nickel titanium instruments present high levels of flexibility and elasticity, which are desirable properties for root canal preparation of curved canals [1, 2]. NiTi instruments manufactured from conventional NiTi alloy have an austenite phase at higher temperatures, and when submitted to cooling or the application of mechanical stresses, they can change to the martensite phase. This phase transition allows for greater flexibility and is called martensitic transformation [3].

Since the introduction of the NiTi alloy for the manufacture of endodontic instruments, several different engine-driven rotary and reciprocating systems have been developed and made

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commercially available [1, 2, 4, 5]. Manufacturers have produced several NiTi instruments with different cross-sectional designs, tapers, tip sizes, surface treatment kinematics and heat treatments with the purpose of improving their mechanical properties and clinical performance [1–7]. The aim of using heat treatment technology (e.g. M-Wire; Dentsply Tulsa Dental Specialties, Tulsa, OK, USA), controlled memory wire (CM; Coltene, Cuyahoga Falls, OH, USA), Blue technology (Dentsply Tulsa Dental Specialties), Gold technology (Dentsply Tulsa Dental Specialties) and R-phase wire (SybronEndo, Orange, CA, USA) is to modify the transitional temperature of martensitic transformation, favouring a high percentage of the martensitic phase. Previous reports have indicated that a high percentage of the martensitic phase increases the flexibility of heat-treated NiTi instruments and improves the performance in terms of cyclic fatigue resistance [1, 6–9]. The heat treatment effect is dependent on time, temperature, processing history and amount of prior cold work, which will promote different mechanical properties for NiTi instruments, including the transitional temperature of martensitic transformation [2, 10].

Reciprocating motion has been widely used in reciprocating single file systems and has been shown to be safer for the root canal preparation of curved canals by reducing cyclic and torsional fatigue [11–13]. In addition, the reciprocating instruments are manufactured with heat treatment technology that favours better cyclic fatigue resistance than that of instruments manufactured from conventional NiTi [11, 12, 14]. The Reciproc Blue (VDW GmbH, Munich, Germany) and WaveOne Gold (Dentsply Tulsa Dental Specialties, Tulsa, OK, USA) systems are manufactured by means of Blue and Gold technologies, respectively [15–17]. Previous studies on their cyclic fatigue resistance have shown that Reciproc Blue presented higher cyclic fatigue resistance than WaveOne Gold at room temperature [15–17].

Recently, a new reciprocating system—the X1 Blue file—was introduced to the market. This system has three instruments with #20, #25 and #40 tip sizes and 0.06 taper (MKLife, Porto Alegre, RS, Brazil), presents a convex triangular cross section and is manufactured with heat treatment similar to that of Blue technology. There is no report regarding the mechanical properties of this system.

Several previous studies have addressed the cyclic fatigue resistance of NiTi instruments at room temperature [4, 5, 7, 11, 14–17]. However, some authors have shown that body temperature drastically affects the flexural resistance of NiTi instruments because it is capable of modifying the transformation temperatures of the NiTi [18–20]. Therefore, the cyclic fatigue test should be performed close to body temperature to ensure relevant results and simulate the flexural behaviour at temperatures inside the root canal [19, 20].

The purpose of this study was to evaluate the impact of body temperature on the cyclic fatigue resistance of different NiTi alloys used for the manufacturing of Reciproc Blue, X1 Blue File and WaveOne Gold systems. The null hypotheses were as follows: (1) there would be no difference in the cyclic fatigue resistance among the instruments at room temperature and body temperature, and (2) the body temperature would not affect the cyclic fatigue resistance of the instruments.

Materials and methods

Cyclic fatigue analysis

A total of 60 NiTi instruments (25 mm length) were selected for this study. The sample calculation was performed using G*Power v3.1 for Mac (Heinrich Heine, University of Düsseldorf) and the Wilcoxon and Mann-Whitney tests of the *t* test family. An alpha-type error of 0.05, a beta power of 0.95 and an N2/N1 ratio of 1 were also stipulated. A total of 10 instruments for each group were used as the ideal size to note significant differences. Every instrument was inspected for defects or deformities before being tested under a stereomicroscope (Carls Zeiss, LLC, EUA) at × 16 magnification; none were discarded. All files used were 25 mm long, and 10 instruments of each brand were used for cyclic testing.

For each reciprocating system, the tip size #25 instrument was selected as follows: Reciproc Blue R25 (RB 25.08), WaveOne Gold Primary (WOG 25.07), and X1 Blue File 25.06 (X1 25.06). All instruments are commercialised in sterile blister pack by the manufacturers. Therefore, the instruments were not sterilised previously the cyclic fatigue test. The instruments were tested for cyclic fatigue at room (20° ± 1 °C) and body (37° ± 1 °C) temperatures using a water bath. The instruments were used coupled to a VDW Silver Motor (VDW, Munich, Germany) connected to the cyclic fatigue device. The pre-set programs were selected according to the manufacturers' recommendations. RB 25.08 was operated with the "Reciproc ALL" program (300 rpm) and WOG 25.07 and X1 25.06 with the "WaveOne ALL" program (350 rpm).

The cyclic fatigue test was performed in a custom-made device that simulated an artificial canal made of stainless steel, with a 60° angle of curvature and a 5-mm radius of curvature located 5 mm from the tip of the instrument (Fig. 1a). The curvature of the artificial canal was fitted onto a cylindrical guide made of the same material (radius 5 mm). There was an external arch that had a 1-mm-deep groove that served as a guide path for the instruments, which kept the instruments on the curvature, allowing them to rotate freely during the test. The device allowed an accurate and reproducible position of the curvature to be established for all the instruments (Fig. 1b). In addition, there were markings with the angles chosen for the test; thus, the iron base could always be fixed in the chosen position and be vertically and horizontally fixed (Fig. 1c).

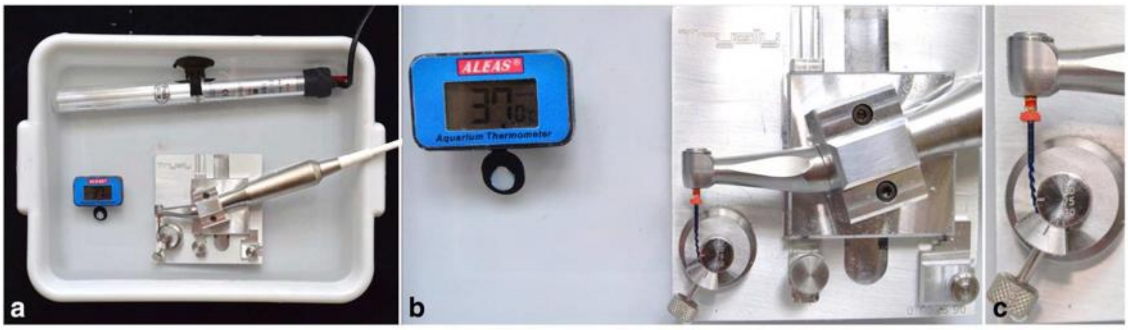

Fig. 1 **a** Experimental setup showing the reciprocating motor, cyclic fatigue test device, aquarium thermostat and aquarium digital thermometer in a plastic container filled with water. **b** Cyclic fatigue test device submerged in a water bath at 37.0 °C. **c** At greater magnification, an experimental instrument in an artificial canal with an angle of curvature of 60° and a radius of 5 mm

The cyclic fatigue tests were performed at room (20° ± 1 °C) and body (37° ± 1 °C) temperatures using a water bath, as in previous studies [18, 19]. At room temperature, the cyclic fatigue test system was submerged in a plastic container filled with 400 ml of deionised cooled water at 20 °C. A digital thermometer (Aleas, Guangdong, China) was used to measure the temperature of the water, and the water temperature was allowed to equilibrate for a few minutes before initiating testing for each group. For the body temperature experiments, the cyclic fatigue test was performed in the same plastic container filled with 400 ml of deionised water at 24 °C. However, an aquarium thermostat (Hopar, Guangdong, China) was submerged in the water a few minutes before starting the testing of each file to achieve the desired temperature (37° ± 1 °C) (Fig. 2). During all tests, the temperatures were monitored by an aquarium digital thermometer.

The time to fracture was recorded using a digital chronometer, and data were also captured by a digital camera to ensure accuracy in the detection of the time of fracture in seconds. In addition, the time to fracture (in seconds) was multiplied by the number of rotations per minute/60 (number of rotation per seconds) to obtain the number of cycles to fracture (NCF) for each instrument. The data were then statistically analysed using one-way ANOVA and Tukey's tests for inter-group comparison at the two temperatures tested and for the reduction in cyclic fatigue time (in percentage) at body temperature. For intra-group comparison at different temperatures, the unpaired t test was used. The significance level was set at 5%.

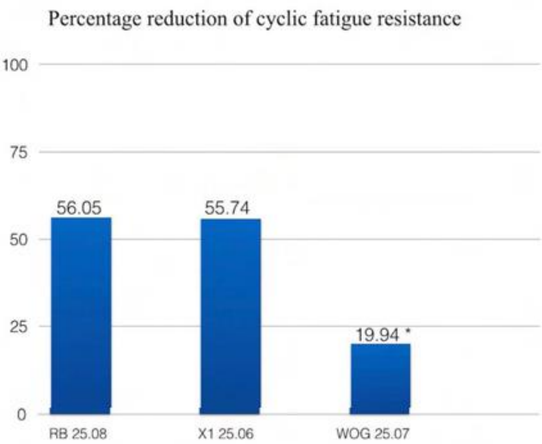

Fig. 2 Percentage reduction of cyclic fatigue resistance of the instruments when exposed at 37.0 °C

Results

The mean and standard deviations of the cyclic fatigue resistance (in time and number of cycles) at room temperature and body temperature for each instrument are presented in Table 1. In addition, the percentage of reduction in the cyclic fatigue at body temperature was calculated, and these data are also shown in Fig. 2.

The cyclic fatigue test at room temperature showed that RB 25.08 and X1 25.06 presented significantly higher TTF and NCF than WOG 25.07 ($P < 0.05$). No difference was found between RB 25.08 and X1 25.06. At body temperature, the TTF and NCF were significantly reduced for all groups compared with room temperature ($P < 0.05$). Regarding the TTF, there was a significant difference between RB 25.08 and WOG 25.07 ($P < 0.05$). No difference was found between RB 25.08 and X1 25.06. The comparison of NCF showed that there was no significant difference among the groups ($P > 0.05$).

The WOG 25.07 presented the lowest percentage reduction of cyclic fatigue in comparison with RB 25.08 and X1 25.06 ($P < 0.05$). No difference was found between RB 25.08 and X1 25.06.

Discussion

Cyclic fatigue is one of the main causes of NiTi engine–driven instrument fractures [11, 12, 21]. Several improvements have

Table 1 Mean values of time (in seconds) and number of cycles (NCF) of instruments tested at different temperatures

Instruments	Cyclic fatigue (s)				Cyclic fatigue (NCF)			
	20 °C		37 °C		20 °C		37 °C	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD
RB 25.08	$694.30^{a,A}$	55.79	$303.60^{a,B}$	21.90	$3473.0^{a,A}$	278.9	$1521.0^{a,B}$	109.4
X1 25.06	$637.80^{a,A}$	53.75	$282.00^{a,b,B}$	32.94	$3726.0^{a,A}$	322.0	$1647.0^{a,B}$	192.1
WOG 25.07	$328.80^{b,A}$	45.57	$262.30^{b,B}$	31.20	$1919.0^{b,A}$	265.6	$1532.7^{a,B}$	182.4

SD, standard deviation

Different superscript lowercase letters in columns represent significant differences among the groups ($P < 0.05$). Different superscript uppercase letters in raw represent significant differences of the instruments in different temperatures ($P < 0.05$)

been introduced in instrument design and manufacturing methods to improve their mechanical properties [1, 4, 7, 16, 17]. Previous studies have shown that Reciproc Blue was more cyclic fatigue resistant than WaveOne Gold at room temperature [15–17]. There is no report of the cyclic fatigue resistance of the X1 Blue file. Recently, some authors demonstrated that the environmental temperature affects the cyclic fatigue resistance of NiTi instruments [18–20]. Therefore, the aim of this study was to evaluate the impact of body temperature on the cyclic fatigue resistance of reciprocating instruments manufactured by different heat treatment technologies.

The methodology used in this study was similar to that of previous studies [18, 19], which allowed for the evaluation of the cyclic fatigue resistance of endodontic instruments under different temperature conditions. The methodology used in this study did not use synthetic oil to lubricate the artificial canal, as previous studies [18–21]. The friction between the instruments and artificial canal walls could lead to increase in the temperature due to the friction between the instruments and artificial canal, increasing the risk of crack formation and/or growth [21]. Cheung and Darvell [22] showed that the aqueous media did not affect the cyclic fatigue test. Additionally, the authors affirmed that aqueous media simulate irrigating solutions more closely and avoid to increase the temperature. Future studies should be performed to evaluate the best method to reduce the friction between the NiTi instruments and artificial stainless steel canals when the cyclic fatigue device is submerged in a water bath. Despite the limitation of the methodology used in this study, several studies have been using a water bath to simulate the body temperature [18–20]. The intracanal temperature ranges from 31 to 35 °C [23, 24], different from the room temperature of several previous studies [4, 7, 8, 11–16]. Therefore, this study was performed with strict temperature control to ensure accurate results.

The first results of this study showed that RB 25.08 and X1 25.06 had higher TTF and NCF values than WOG 25.07 at room temperature ($P < 0.05$). At body temperature, there was significant difference only between RB 25.08 and WOG 25.07 when comparing the TTF ($P < 0.05$). When the NCF was taken into consideration, there was no significant difference among the groups ($P > 0.05$). Thus, the first null hypothesis was partially rejected. The instruments used in this study had the same tip sizes (#25). However, the tapers differed among them. The Reciproc Blue had a taper of 0.08 mm/mm, WaveOne Gold a nominal taper of 0.07 mm/mm and X1 Blue File a nominal taper of 0.06 mm/mm along the first 3 mm from the tip. Previous studies have shown that instruments with lower taper should ensure higher cyclic fatigue time [7, 25]; nevertheless, our results showed that the Reciproc Blue group presented higher TTF than X1 25.06 and WOG 25.07. Therefore, other variables, such as cross-sectional design, diameter of the core, heat treatments and type of reciprocating motion, should be considered in the outcomes of this study.

The results of this study were probably caused by the different cross-sectional designs and types of NiTi alloy of the instruments, which affected the mechanical properties of the NiTi instruments [1, 7, 26]. The RB 25.08 had S-shaped cross sections with two cutting edges, the X1 Blue file a convex triangular cross section and the WOG 25.07 a parallelogram-shaped cross section. These different features among the instruments probably provided a different metal mass volume. Previous studies have shown that a larger metal mass volume at the maximum stress point of NiTi instruments affected cyclic fatigue resistance [7, 11, 22, 25], which could account for the difference in the cyclic fatigue lifetimes of the instruments.

The heat treatments of NiTi alloys have strong influences on the martensitic/austenitic transformation behaviour, favouring a different arrangement of the crystalline structure and a higher percentage of martensitic transformation [1, 4, 9, 11, 14]. Some authors have shown that a higher percentage of martensitic phase in the NiTi alloy promoted more flexibility and greater fatigue resistance [1, 4, 6, 9]. Our results showed that WOG 25.07 presented the lowest TTF and NCF compared with RB 25.08 and X1 25.06 at room temperature ($P < 0.05$) and lower TTF than RB 25.08 at body temperature ($P < 0.05$). The results of this study agree with those of previous studies, which showed that RB 25.08 had higher TTF than WOG 25.07 [15–17]. The results of X1 25.06 were similar to those of RB 25.08. Despite the difference in taper and cross-sectional design between them, the similar heat treatments could explain our results. The Blue technology of RB 25.08 and X1 25.06 could probably result in different martensitic phase transformations at the two temperatures tested compared to the Gold technology used for WOG 25.07, providing differences in flexibility and dissipation of the energy required for crack formation and/or propagation during cyclic fatigue testing [1].

Previous studies have shown that the cyclic fatigue resistance of NiTi instruments was drastically affected at body temperature [18–20]. The results of this study showed that the three reciprocating instruments exhibited reduced cyclic fatigue resistance at 37 °C. Therefore, our second null hypothesis was rejected. Despite the differences in cross-sectional design and taper among the instruments, the results at body temperature showed that the heat treatment may play a role in the results of this study. WOG 25.07 presented similar results of cyclic fatigue time to failure as X1 25.06. In addition, WOG 25.07 presented the lowest reduction in cyclic fatigue resistance compared with RB 25.08 and X1 25.06. There was no difference between RB 25.08 and X1 25.06 regarding the cyclic fatigue time to failure and the reduction in cyclic fatigue resistance. The reduction in cyclic fatigue resistance of the three reciprocating instruments at body temperature could be related to the differences in the martensitic transformation temperature between the Blue and Gold treatments [9, 18, 19], which could have induced a different degree of austenitic phase at body temperature.

Data from this study suggested that Blue technology instruments were more affected than Gold technology instruments at body temperature. The crystal lattice of RB 25.08 and X1 25.06 probably changed more abruptly at body temperature than that of WOG 25.07, which could explain the lower reduction in cyclic fatigue resistance of WOG 25.07. The austenite finishing temperature of Blue technology is lower (33.71–38 °C) [18, 19] than that of Gold (50.1–51.8 °C) [9]. At body temperature, RB 25.08 and X1 25.06 probably presented a lower percentage of martensitic phase than WOG 25.07, thereby decreasing the flexibility and the cyclic fatigue resistance. Our results support those of previous investigations in which the authors showed a reduction of the cyclic fatigue time of heat-treated NiTi instruments exposed to body temperature [18–20]. These results are clinically relevant because the mechanical behaviour of the NiTi instruments could be affected during root canal treatment.

Despite our results showed that WOG 25.07 presented lowest TTF at both temperatures tested, when the NCF is taken in consideration, there was no significant difference among the instruments at body temperature ($P > 0.05$). This fact could be explained because of the different speeds between the "Reciproc ALL" and "WaveOne ALL" programs and percentage reduction in fatigue resistance. The "Reciproc ALL" mode works at 300 rpm, while the "WaveOne ALL" operates at 350 rpm [27]. The higher rotational speed induces greater number of cycles and more compressive and tensile stresses per second, which could favour instrument fracture [26–29]. However, due to a significant greater reduction of TTF of RB 25.08 and X1 25.06 at body temperature, the WOG 25.07 presented similar NCF than other groups ($P > 0.05$). Therefore, if we would consider only the NCF at body temperature, we could affirm that these three instruments tested presented similar cyclic fatigue resistance.

Conclusion

In conclusion, within the limitations of this study, the body temperature treatment caused a marked reduction in the cyclic fatigue resistance for all reciprocating instruments tested. The RB 25.08 and X1 25.06 systems presented similar results at both temperatures tested. However, WOG 25.07 presented the lowest percentage reduction in fatigue resistance at body temperature.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflicts of interest.

Ethical approval This article does not contain any studies with human participants or animals performed by any of the authors.

Informed consent For this type of study, formal consent is not required.

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2.2 ARTICLE 2 – EVALUATION OF CYCLIC AND TORSIONAL FATIGUE RESITANCE OF FOUR ROTARY INSTRUMENTS WITH SIMILAR CROSS-SECTION AND DIFFERENT THERMAL TREATMENTS

The second article presented in this thesis was published in *Dental Press Endodontics*.

artigo inédito

Avaliação da resistência à fadiga cíclica e torcional de quatro instrumentos rotatórios de NiTi com secções transversais semelhantes e diferentes tratamentos térmicos

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RESUMO

Objetivo: O objetivo do presente trabalho foi avaliar a resistência à fadiga cíclica e torcional de instrumentos rotatórios de níquel-titânio com secções transversais semelhantes e confeccionados com diferentes tipos de tratamentos térmicos: Hyflex CM (HCM 25/.06), Vortex Blue (VB 25/.06), Sequence Rotary File (SRF 25/.06) e EdgeSequel (EDF 25/.06) (n=20). **Métodos:** O teste de fadiga cíclica avaliou o tempo e o número de ciclos para a fratura (NCF) dos instrumentos em canal artificial com 60° de curvatura e 5 mm de raio (n=10). O teste de torção avaliou o torque máximo e a deflexão angular para a fratura dos 3 mm da ponta dos instrumentos (n=10). Após o teste de torção e de fadiga cíclica, os instrumentos foram avaliados em microscopia eletrônica de varredura (MEV) (n=10). Os dados foram analisados empregando-se os testes ANOVA e de Tukey, sendo selecionado um nível de significância de 5%. **Resultados:** HCM apresentou o maior tempo e NCF entre todos os instrumentos avaliados ($p<0,05$). O SRF apresentou um tempo semelhante ($p>0,05$) e menor NCF ($p<0,05$) do que o VB. Em relação ao teste torcional, o HCM apresentou menor valor de torque e maior deflexão angular entre os grupos avaliados ($p<0,05$). Não houve diferenças significativas entre VB, SRF e EDF em relação à deflexão angular ($p>0,05$). **Conclusão:** O HCM apresentou maior resistência à fadiga cíclica e deflexão angular. Entretanto, o VB apresentou maior resistência torcional para a fratura.

Palavras-chave: Instrumentos odontológicos. Endodontia. Níquel. Titânio.

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Introdução

Os instrumentos mecanizados de níquel-titânio (NiTi) têm sido amplamente utilizados para o preparo de canais curvos, devido à sua flexibilidade e elasticidade, proporcionando segurança durante o preparo[1,2]. No entanto, a fratura inesperada desses instrumentos pode ocorrer devido a diversos fatores. As causas mais comuns são a fadiga cíclica e torcional[3,4].

A fratura por fadiga cíclica ocorre devido ao estresse de tração e compressão no ponto máximo de flexão durante o preparo de um canal curvo[4,5]. A fadiga por torção ocorre quando a ponta do instrumento se trava nas paredes do canal radicular e ele continua seu movimento de rotação, sendo comum em anatomias constrictas[4,5]. Por essa razão, os fabricantes desenvolveram instrumentos com diversos *designs* (conicidades, secções transversais e diâmetros de ponta) e tratamentos térmicos da liga de NiTi, assegurando uma melhora nas propriedades mecânicas e reduzindo o risco de fratura[1-6].

Estudos prévios demonstraram que a resistência a fadiga cíclica e torcional dos instrumentos de NiTi é diretamente afetada pelos diferentes *designs*[1,2,6-10]. Portanto, pode-se especular que instrumentos com *designs* semelhantes devem apresentar propriedades mecânicas similares. Por outro lado, o tratamento térmico do NiTi proporciona uma alteração nas propriedades mecânicas, sendo necessário levar em consideração a liga metálica empregada na fabricação desses instrumentos[9,11-15].

Os tratamentos térmicos são capazes de controlar a temperatura de transformação e induzir uma reestruturação dos átomos da liga metálica[1,2,10-12]. Sendo assim, dependendo do tipo de tratamento térmico, haverá diferentes porcentagens de fase austenita, martensita ou Fase R, afetando diretamente a flexibilidade e resistência à fratura dos instrumentos[1,6,10-12].

O sistema rotatório Hyflex (HCM; Coltene, Whaladent, Suíça) é um sistema composto por instrumentos que apresentam secção triangular convencional, *taper* fixo e são fabricados com um tratamento térmico denominado Memória Controlada (CM-Wire, Ds Dental Johnson City, Tennesse, EUA)[16]. Diversos estudos prévios demonstraram a alta flexibilidade e resistência à fadiga cíclica dos seus instrumentos quando comparados com instrumentos confeccionados com NiTi convencional e outros tipos de ligas tratadas termicamente[2,5,16,17].

O sistema rotatório EdgeSequel Sapphire (EdgeEndo, Albuquerque, Tennesse, EUA) é composto por instrumentos com secção triangular, *taper* fixo e fabricado com um tratamento térmico denominado Fire-Wire. De acordo com o fabricante, esse tratamento é semelhante à tecnologia de memória controlada, favorecendo alta flexibilidade aos instrumentos[18]. Não há qualquer estudo comparando as propriedades mecânicas dos instrumentos desse sistema com o sistema Hyflex CM.

O sistema Vortex Blue (VB; Dentsply-Sirona, EUA) é um sistema rotatório confeccionado com secção triangular e tratamento térmico Blue[13]. Estudos prévios demonstraram que os instrumentos VB apresentam menor resistência à fadiga cíclica que os instrumentos HCM em temperatura ambiente[19].

Recentemente, um novo sistema rotatório foi introduzido no mercado nacional, o Sequence Rotary File (Mk Life, RS, Brasil). Esse sistema é composto por quatro instrumentos (15.04, 20.06, 25.06 e 35.04) com conicidade fixa e tratamento térmico semelhante ao do Vortex Blue. O único instrumento que não apresenta secção triangular é o 15.04, sendo quadrangular. De acordo com o fabricante, o tratamento térmico favorece uma coloração azulada, devido à formação de uma camada de óxido de titânio em sua superfície, e confere alta flexibilidade à liga.

Embora a literatura tenha demonstrado o impacto dos diferentes tratamentos térmicos nas propriedades mecânicas dos instrumentos mecanizados de NiTi, há uma escassez de estudos avaliando a resistência à fadiga cíclica e torcional de instrumentos que apresentem como principal diferença apenas a liga metálica. Portanto, o objetivo do presente estudo foi avaliar a resistência à fadiga cíclica e torcional de instrumentos rotatórios com *designs* semelhantes (conicidade e secção transversal) e diferentes tipos de tratamentos térmicos. As hipóteses nulas testadas foram: 1) não há diferença na resistência à fadiga cíclica entre os instrumentos avaliados; e 2) não há diferença na fadiga torcional entre os instrumentos avaliados.

Material e Métodos

O cálculo amostral foi realizado antes do teste mecânico, usando o G*Power v3.1 para Mac (Heinrich Heine, University of Düsseldorf, Alemanha) e selecionando o teste Wilcoxon-Mann-Whitney, da

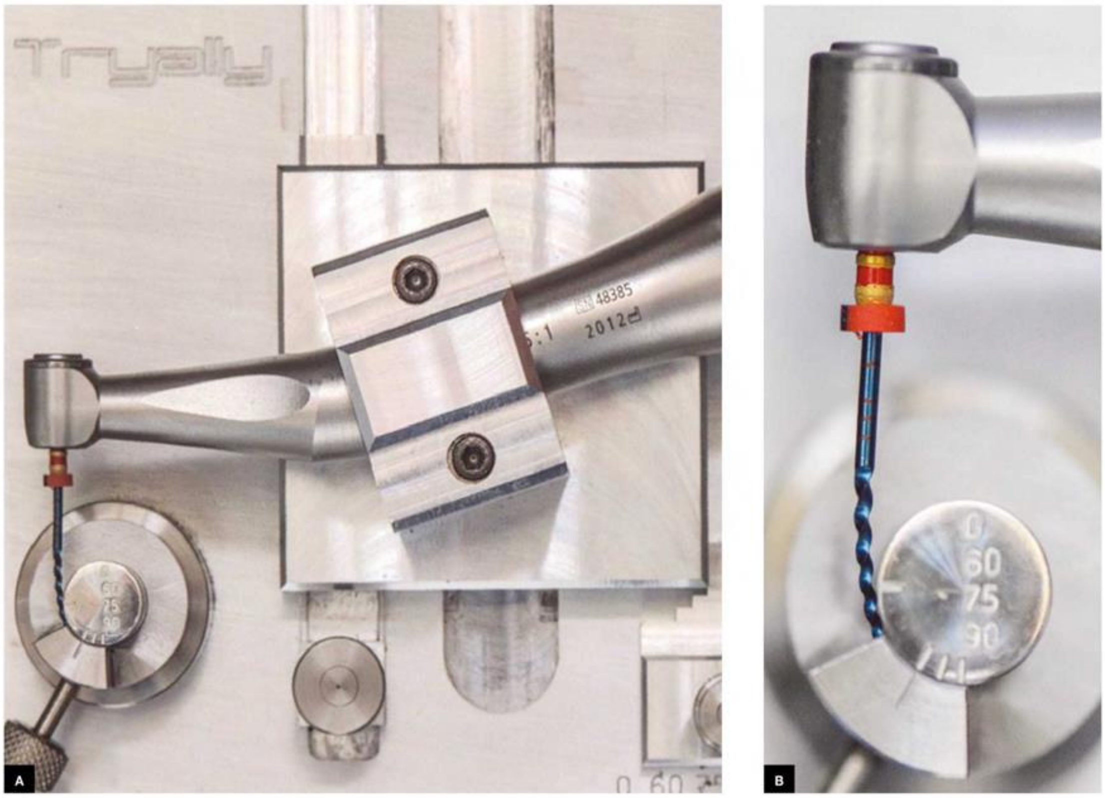
família do teste *t*. Foram, também, estipulados o erro alfa em 0,05, o poder do beta em 0,95 e uma proporção N2/N1 de 1. O teste mostrou um total de 10 amostras para cada grupo como o tamanho ideal para identificar diferenças significativas.

Foram utilizados 80 instrumentos de quatro sistemas rotatórios (n=20): Hyflex CM (HCM; #25/.06); Vortex Blue (VB; #25/.06); Sequence Rotary File (SRF; #25/.06); e EdgeSequel (EDF; #25/.06).

Teste de fadiga cíclica

O teste estático de fadiga cíclica foi realizado em um dispositivo feito sob medida, simulando um canal artificial, feito de aço inoxidável, com ângulo de curvatura de 60°, raio de curvatura de 5mm e com um 1 mm de profundidade, como previamente descrito[20] (Fig. 1). O teste de fadiga cíclica foi realizado à temperatura ambiente (21±1°C).

Dez instrumentos rotatórios de cada sistema foram ativados por um contra-ângulo 6:1 (Sirona Dental Systems GmbH, Bensheim, Alemanha) acoplado em um motor endodôntico (Reciproc Silver, VDW, Alemanha) seguindo as orientações dos fabricantes. Os instrumentos HCM, VB e EDF foram ativados a uma velocidade de 500 rpm, enquanto o sistema SRF foi usado a uma velocidade de 400 rpm. Durante todo o experimento, o canal artificial era lubrificado com óleo sintético, para prevenção de aquecimento (Super



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Os tratamentos térmicos são capazes de controlar a temperatura de transformação e induzir uma reestruturação dos átomos da liga metálica[1,2,10-12]. Sendo assim, dependendo do tipo de tratamento térmico, haverá diferentes porcentagens de fase austenita, martensita ou Fase R, afetando diretamente a flexibilidade e resistência à fratura dos instrumentos[1,6,10-12].

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O sistema rotatório EdgeSequel Sapphire (EdgeEndo, Albuquerque, Tennesse, EUA) é composto por instrumentos com secção triangular, *taper* fixo e fabricado com um tratamento térmico denominado Fire-Wire. De acordo com o fabricante, esse tratamento é semelhante à tecnologia de memória controlada, favorecendo alta flexibilidade aos instrumentos[18]. Não há qualquer estudo comparando as propriedades mecânicas dos instrumentos desse sistema com o sistema Hyflex CM.

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Foram utilizados 80 instrumentos de quatro sistemas rotatórios (n=20): Hyflex CM (HCM; #25/.06); Vortex Blue (VB; #25/.06); Sequence Rotary File (SRF; #25/.06); e EdgeSequel (EDF; #25/.06).

Teste de fadiga cíclica

O teste estático de fadiga cíclica foi realizado em um dispositivo feito sob medida, simulando um canal artificial, feito de aço inoxidável, com ângulo de curvatura de 60°, raio de curvatura de 5mm e com um 1 mm de profundidade, como previamente descrito[20] (Fig. 1). O teste de fadiga cíclica foi realizado à temperatura ambiente (21±1°C).

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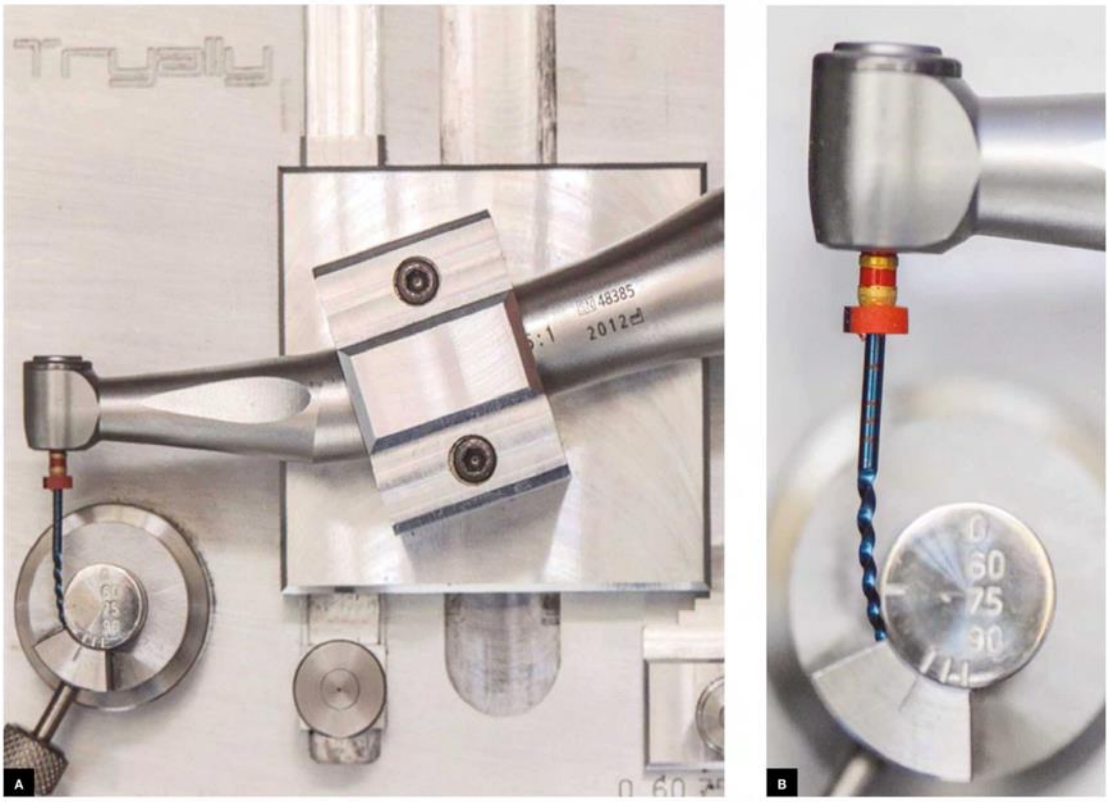

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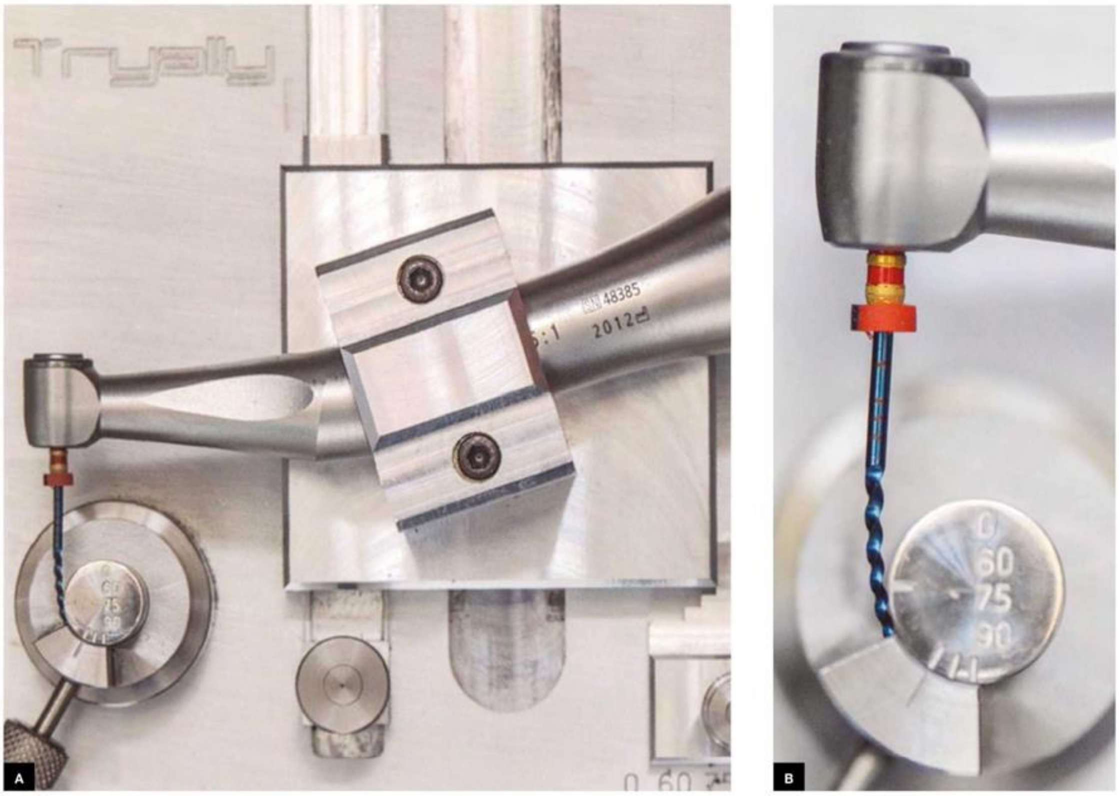

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3 Discussion

3 DISCUSION

During the last decades the manufacturers have been struggling to develop new manufacturing methods, thermal treatments and instruments with different designs to improve the shaping ability, clinical performance and reduce de risk of instruments separation (PEDULLA *et al.*, 2016; GAVINI *et al.*, 2018; ALCALDE *et al.*, 2018, ZUPANC *et al.*, 2018). The cyclic and torsional fatigue is the most commom cause of instruments separation (SATAPAN *et al.*, 2000; PEDULLA *et al.*, 2016). Thus, cyclic and torsional fatigue tests are widely used to evaluate the mechanical properties of NiTi engine-driven instruments (PLOTINO *et al.*, 2014). These mechanical tests aimed to cause cyclic and torsional stress, simulating a mechanical stress caused during a curved or constricted canal, assisting tostipulate how would be the mechanical behavior in these clinical situations (HÜLSMANN *et al.*, 2019).

The cyclic fatigue apparatus used in this both studies was previous reported by several authors (MARKS DUARTE *et al.*, 2018; ALCALDE *et al.*, 2018; ALCALDE *et al.*, 2020; WEISSHEIMER *et al.*, 2020). Our study utilized the static instead of the dynamics model due to the static method decrease some biases, such as amplitude of axial motions and speed, wich are completely subjective in a clinical situation because their reproduciton is manually controlled (ALCALDE *et al.*, 2018). Also, depending on the design of the tube or artificial groove, torsional stress can be created, which could compromisse to distinguish the tupe of fatigue occured, as previously pointed out by Dedrich & Zakariasen (1986) and reinforced for Hülsmann *et al.* (2019). Therefore, in both studies used the static model because dynamics seems to be a more sensitive method and could create other variables beyond the type of the instrument.

It is important to emphasize that there are no specifications or international standards for the evaluation of cyclic fatigue resistance in NiTi instruments. According to Plotino *et al.*, (2017), the artificial canal should reproduce the instruments size and taper to ensure repeatable canal trajectory in terms of radius and angle of curvature. However, this is not possible in our studies because the testes instruments present different tapers. Therefore, to standardize the testing conditions, the same tapered artificial canal was used for all groups

(60° of curvature, 5 mm of radius of curvature, 0.40 mm of diameter at the most apical portion, 0.06 mm/mm taper).

Another methodological point that needs to be adressed is related to temperature conditions during the cyclic fatigue. Although the current studies demonstrated that the cyclic fatigue test should be performed simulating the root canal temperature (VASCONCELOS *et al.*, 2016; JAMLEH *et al.*, 2016; DOSANJH *et al.*, 2017), the first study was performed at 36±1°C while the second at 20±1°C. The difference between then can be explained due to the second study was previously performed and the method to simulating of body temperature was developed during the second study. This fact is the only difference, because the same apparatus, canal and radius of curvatures were used.

In the second study was used the torsional test to evalaute the maxium torsional strength and angular deflection supported by the instruments before separation. The method used was according the ISO Standadr 3630-1 and has been previously reported by several authors (ALCALDE *et al.*, 2017, 2018, 2020). The 3 mm of the tip was fastened and rotation in clockwise direction wa set for all instruments. The the mm of the tip was chosen because atthis point the instruments is more suspectible to fracture than at 5 mm (ÇAPAR *et al.*, 2015). Also, the torcional test was performed at room temperature (20±1°C) because it seems that the different temperature did not affect the torsional properties of NiTi instruments (SILVA *etal.*, 2019).

The first result of the first study showed that Reciproc Blue (RB) 25.08 and X1 Blue File (X1) 25.06 had higher cyclic fatigue resistance values than WaveOne Gold (WOG) 25.07 at room and body temperature ($P<0.05$). The instruments used in this study had the same tip sizes (#25). However, the tapers differed among them; The Reciproc Blue had a taper of 0.08 mm/mm, WaveOne Gold had a nominal taper of 0.07 mm/mm and X1 Blue File a nominal taper of 0.06 mm/mm, along the first 3 mm from the tip. Previous studies have shown that instruments with lower taper should ensure lower metal mass volume and higher cyclic fatigue time (KESKIN *et al.*, 2017; ALCALDE *et al.*, 2018; SILVA *et al.*, 2018); nevertheless, our results showed that Reciproc Blue group presented better results than X1 25.06 and WOG 25.07. Therefore, other variables such as cross-section design diameter of core and heat treatments have impacted in the outcomes of this study.

The RB 25.08 had S-shaped cross-sections with 2 cutting edges; the X1 Blue file a convex triangular cross-section; and WOG 25.07 had a parallelogram-shaped cross-section. These different features among the instruments probably provided a different metal mass volume. Previous studies have shown that a larger metal mass volume at the maximum stress point of NiTi instruments affected cyclic fatigue resistance (KIM *et al.*, 2012; KAVAL *et al.*, 2016; ÇAPER *et al.*, 2015; ALCALDE *et al.*, 2017; SILVA *et al.*, 2018; ALCALDE *et al.*, 2020), which could concur with the difference in the cyclic fatigue lifetimes of the instruments.

The heat treatments of the NiTi alloys have strong influences on martensitic/austenitic transformation behavior, which favor a different arrangement of the crystalline structure and a higher percentage of martensitic transformatin, consequently, high flexibility and greater resistance to fatigue (GUTTMAN *et al.*, 2012; LOPES *et al.*, 2013; SHEN *et al.*, 2013; ZUPANC *et al.*, 2018; GAVINI *et al.*, 2018). Our results showed that WOG 25.07 presented the lowest cyclic fatigue resistance when compared with RB 25.08 and X1 25.06 at room temperature ($p<0.05$); and at body temperature, lower cyclic fatigue resistance than RB 25.08 ($p<0.05$). The results of this study were in agreement with the findings of some authors, which showed that RB 25.08 had higher cyclic fatigue resistance than WOG 25.07 (KESKIN *et al.*, 2017; ALCALDE *et al.*, 2018; SILVA *et al.*, 2018). The results of X1 25.06 were similar to those of RB 25.08. Despite the difference in taper and cross-sectional design between them, the similar heat treatments could explain our results. The Blue technology of RB 25.08 and X1 25.06 could probably result in different martensitic phase transformations than those of Gold used in WOG 25.07 at the two temperatures tested, providing differencesin flexibility and dissipation of the energy required for crack formation and/or propagation during cyclic fatigue testing (GUTTMAN *et al.*, 2012; SHEN *et al.*, 2013; PEDULLA *et al.*, 2016; ZUPANC *et al.*, 2018).

Previous studies have shown that the cyclic fatigue resistance of NiTi instruments was drastically affected at room temperature (VASCONCELOS *et al.*, 2016; JAMLEH *et al.*, 2016; DOSANJH *et al.*, 2017). The results of this study showed that three reciprocating instruments reduced the cyclic fatigue resistance at 37°C. Despite the differences in cross- sectional design and taper among the instruments, the results at body temperature showed thatthe heat treatment may play role in the results of this study. The WOG 25.07 presented similarresults of cyclic fatigue time to failure with X1 25.06. In addition, WOG 25.07 presented the lowest reduction in cyclic fatigue resistance when compared with RB 25.08 and X1 25.06.

There was no difference between RB 25.08 and X1 25.06 regarding the cyclic fatigue time to failure and reduction in cyclic fatigue resistance. The reduction in cyclic fatigue resistance of these instruments at body temperature could be related to the differences in temperature of martensitic transformation between Blue (38°C) and Gold treatment (50°C) (HIEAWY *et al.*, 2015; VASCONCELOS *et al.*, 2016; DOSANJH *et al.*, 2016), which could have induced a different degree of austenitic phase at body temperature.

Data from this study suggested that Blue technology was more affected than Gold at body temperature. The crystal lattice of RB 25.08 and X1 25.06 probably changed more abruptly at body temperature than that of WOG 25.07, which could explain the lower reduction in cyclic fatigue resistance of WOG 25.07. The austenite finishing temperature of Blue technology is lower (33.71- 38°C) (VASCONCELOS *et al.*, 2016; DOSANJH *et al.*, 2016) than that of Gold (50.1-51.8°C) (HIEAWY *et al.*, 2015). At body temperature the RB 25.08 and X1 25.06 probably presented a lower percentage of martensitic phase than WOG 25.07, thereby decreasing the flexibility and the cyclic fatigue resistance. Our results support those of previous investigations in which the authors showed a reduction of cyclic fatigue time of heat-treated NiTi instruments exposed to body temperature (HIEAWY *et al.*, 2015; VASCONCELOS *et al.*, 2016; DOSANJH *et al.*, 2016; JAMLEH *et al.*, 2016). These results are clinically relevant because the mechanical behavior of the NiTi instruments could be affected during root canal treatment.

The second study evaluated the cyclic and torsional fatigue resistance of four NiTi rotary instruments with similar cross-section and different thermal treatments (Hyflex CM 25.06, Vortex Blue 25.06, Sequence Rotary File 25.06 and Edge Sequel 25.06). Altought the cyclic fatigue test was performed at room temperature (20±1°C), as previously discussed, the results could stipulate the mechanical behavior of these instruments in curved canals. However, it would be interesting to repeat this test at root canal temperature (20±1°C) to assess if the results would be impact or not as previously discussed at the first study.

The results of the cyclic fatigue teste showed that Hyflex CM (HCM) had the highest cyclic fatigue resistance (time and NCF) when compared with the others groups ($P<0.05$). The Sequence Rotary File (SRF) presented similar time ($P<0.05$) and lower NCF ($P<0.05$) to fatigue than Vortex Blue (VB). The EdgeSequel (EDS) presented the lowest time and NCF when compared with the other groups ($P<0.05$). In this study, all the instruments had the same tip size (#25), taper and similar cross-sectional design, which could ensure similar cyclic fatigue resistance among them. Thus, the thermal treatment of the NiTi alloy should be considered in the outcomes of this study.

The results of HCM and VB in this study were in agreement with those of a previous study (SHIM *et al.*, 2018) that showed HCM 25.06 was more cyclic fatigue resistant than VB 25.06 at room temperature. The thermal treatments modify the martensitic/austenitc transformation behavior, which can induce a higher level of flexibility due to a different arrangement of the crystalline structure and a higher percentage of martensite or R-phase (GUTTMAN *et al.*, 2012; LOPES *et al.*, 2013; SHEN *et al.*, 2013; VASCONCELOS *et al.*, 2016; ZUPANC *et al.*, 2018). In addition, the martensite phase favor slower fatigue crack propagation speed than that of the austenite phase (PEREIRA *et al.*, 2012; SHEN *et al.*, 2013; PEDULLA *et al.*, 2016). Therefore, the different phase transformation of the NiTi probably affected our results.

The controlled memory instruments have a martensite and R-phase (ZUPANC *et al.*, 2018), while Blue treatment of V 25.06 B induces a classic R-phase (VASCONCELOS *et al.*, 2016; PLOTINO *et al.*, 2014). HCM group probably presented higher percentage of martensite phase, which could explain the best performance. The results of VB 25.06 andSRF 25.06 groups could be related to the similar thermal treatment between them. Lastly, EDS 25.06 probably presented a high percentage of austenite phase.

During the cyclic fatigue test all instruments were used in accordance with the manufacturer's recommendations. HCM, VB and EDF were used at 500 rpm for cyclic fatigue test, while SRF, at 400 rpm. The higher rotational speed increases the cyclic fatigue stress due to inducing more compressive and tensile stresses in the NiTi instruments (LOPES *et al.*, 2009). Therefore, if all instruments were used at 500 RPM, probably the results of SRF could be negative affected. The results of EDS group suggest that would be safer to use them at a lower speed than that specified in the manufacturer's instructions, reducing the risk of instrument failures.

The torsional test determined the maximum torque load and distortion angle to failure at the 3 mm of the instrument tip, according ISO Standard 3630-1. HCM 25.06 had the lowest torque load when compared with the other groups ($P<0.05$). VB 25.06 presented the highest torque load in comparison with all the groups ($P<0.05$); no difference was found between SRF 25.06 and EDS 25.06 ($P>0.05$). In relation the distortion angle, HCM 25.06 showed higher angle values than the other groups (P<0.05). There was no difference among VB25.06, SRF 25.06 and EDS 25.06 (P>0.05). The design features (taper, tip size, cross-section and core diameter) of NiTi instruments affected the torsional resistance (WYCOFF; BERZIN,2012; BAEK *et al.*, 2012; ALCALDE *et al.*, 2018; SILVA *et al.*, 2018; ALCALDE *et al.*, 2020). All the instruments used in this study presented similar design features (taper, cross-section and tip size), which should have ensured similar torsional resistance. However, this did not occur.

Previous studies have shown there was an inverse tendency between flexibility and core diameter (ZHANG *et al.*, 2011; BAEK *et al.*, 2012). Thus, based on our results, it might be speculated that HCM presented smaller core diameter. Additionaly, depending on the type of thermal treatment, a different arrangement of the crystal structure could be induced,changing the mechanical properties (SHEN *et al.*, 2013; PEDULLA *et al.*, 2016; ALCALDE *et al.*, 2018). CM-Wire instruments presented low torsional loads and high distortion angle to failure due to the presence of martensite and R-phase, which reduced the elastic modulus and increased the deformation capacity (PEDULLA *et al.*, 2016; ZUPANC *et al.*, 2018; GAVINI *et al.*, 2018). The thermal treatments probably induced different martensite transformation of the NiTi among them. Therefore, the core diameter and thermal treatments played an important role in our results.

The Electron Scanning Microscopy (SEM) analysis was perfored to analyze the topographic features of the instruments after the cyclic and torsional test. After the cyclic fatigue test, the instruments showed crack initiation areas and overload zones, with numerous dimples spread across the fractured surfaces. After the torsional test, the fragments showed concentric abrasion marks and fibrous dimples at the center of rotation.

Althougth the studies have been evaluated the cyclic flexural and torsional fatigue resistance in separated tests, it well known that during root canal preparation the instruments fractures can occur due to a association of both fatigue (SATTAPAN *et al.*, 2000; PEDULLA *et al.*, 2016). Therefore, in future studies, it would be important to develop new methodologies trying to reproduce the real mechanical behaviour of the NiTi instruments during root canal preparation.

The results of both studies study have an important clinical significance, allowing speculate the mechanical behavior of the NiTi instruments on a curved or constricted canal.

Instruments with higher cyclic fatigue resistance indicate that they are much flexible and safe for preparing curved root canals, and probably cause fewer undesirable changes in the root canal anatomy. On the other hand, in cases of constricted canals, it is suitable to use instruments that present greater torsional resistance because they are submitted to higher torsional stress, which could cause plastic deformation or instrument separation.

4 CONCLUSIONS

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In relation the results of the two articles were possible concluded that:

a) The body temperature treatment caused a marked reduction in the cyclic fatigue resistance for all reciprocating instruments tested. The RB 25.08 and X1 25.06 systems presented similar results at both temperatures tested. However, WOG 25.07 presented the lowest percentage reduction in fatigue resistance at body temperature.

b) The thermal treatments of the NiTi are important determinants of the mechanical properties of the NiTi instruments. The HCM demonstrated the highest cyclic fatigue resistance and distortion angle to failure then the others instruments. However, the VB showed highest torque load and lowest anglular deflexion.

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