Comparison of Different Temperatures on Bending Properties of Six Niti Endodontic File Systems

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COMPARISON OF DIFFERENT TEMPERATURES ON BENDING PROPERTIES OF SIX NITI ENDODONTIC FILE SYSTEMS

by

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A Thesis submitted to the Faculty of the Graduate School,
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ABSTRACT

COMPARISON OF DIFFERENT TEMPERATURES ON BENDING PROPERTIES OF SIX NITI ENDODONTIC FILE SYSTEMS

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Marquette University, 2018

Introduction: Manufacturers claim that modern NiTi files with proprietary heat treatment transform at higher temperatures, thus staying more martensite and being more resistant to cyclic fatigue and more flexible. There are some studies comparing the effect of body temperature and room temperature on cyclic fatigue of these newer NiTi files. However, there is not yet a study published for evaluating the relationship between bending properties of NiTi instruments and temperature following the ISO 3630-1 guideline. The objective of this study was to evaluate how temperature affects the bending properties of six different brands of NiTi rotary instruments with different transformation temperature ranges.

Methods: Six commercially available NiTi files were selected for this experiment. The tested files included K3 40/.04 (Sybron Endo, Orange, CA), ProFile Series 29 Green Size 6 (Dentsply Tulsa Dental Specialties), K3XF 40/.04 (Sybron Endo, Orange, CA), Vortex Blue 40/.04 (Dentsply Tulsa Dental Specialties), ProFile Vortex 40/.04 (Dentsply Tulsa Dental Specialties), and HyFlex CM™ 40/.04 (Coltène/Whaledent Inc., Cuyahoga Falls, OH). The Austenite finish temperatures of the files were 9.6 ± 0.5, 17.6 ± 0.6, 24.9 ± 1.1, 35.4 ± 1.2, 45.7 ± 0.9, and 60.3 ± 3.1, respectively. The bending properties of the files were measured using a torsiometer (Sa bri Dental Enterprises, Inc. Downers Grove IL) following ISO 3630-1 guidelines. Twelve of each file type were grouped into 3 groups based on temperatures. Each temperature group had a total of 72 files. Group 1 measured the bending moment (g·cm) at 9±2°C, group 2 at 23±2°C, and group 3 at 35±2°C. The data was statistically analyzed by ANOVA and *post hoc* HSD ($P<0.05$)

Results: For all tested files, the bending moment of the files increased as the temperature rose from 9to 23 to 35°C. At all temperatures, HyFlex CM was significantly more flexible than other files. ProFile Vortex, K3XF, and Vortex Blue showed similar flexibility with each other. They were significantly more flexible than ProFile Series 29, which was significantly more flexible than K3.

Conclusion: Testing temperature and brand of the files were significant independent variables affecting the flexibility of the files.

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CHAPTER 1

INTRODUCTION

For endodontic disinfection to be effective, canal identification, cleaning, and shaping is critical. Mechanical debridement of canals can be achieved through the use of endodontic instruments, such as stainless steel hand files and Nickel-Titanium (NiTi) rotary instruments. NiTi's superior flexibility and torsional properties compared to stainless steel make it possible for it to be used in a rotary handpiece, making canal instrumentation safer and more efficient (Haapasalo et al., 2013). In addition, advancements in thermal treatments such as M-wire, Blue-wire, R phase, and controlled memory technologies can offer improved mechanical properties of flexibility and cyclic fatigue, as compared with conventional NiTi instruments (Shen et al., 2013). Improved flexibility of the file facilitates optimal cleaning and shaping of curved canals with fewer iatrogenic complications, such as ledging, zipping, and transportation (Kuhn et al., 1997).

Of note for the practitioner are the considerations of rotary instrumentation in the clinical setting. Intracanal temperature ranges between 30-35°C (Hemptinne et al., 2015); however, many in vitro studies have been conducted at either room temperature or it was unspecified. Of clinical significance, and where a gap in the literature exists, is in examining the bending properties of different nickel-titanium rotary files at intracanal temperatures.

Manufacturers claim that modern NiTi files with proprietary heat treatment transform at higher temperatures, thus staying more martensite and being more resistant to cyclic fatigue and more flexible. There are some studies comparing the effect of body temperature and room temperature on cyclic fatigue of these newer NiTi files (Dosanjh et al., 2017, Plotino et al., 2017). However, there is not yet a study published for evaluating the relationship between bending properties of NiTi instruments and temperature following the ISO 3630-1 guideline (2008).

The objective of this study was to evaluate how temperature affects the bending properties of six different brands of NiTi rotary instruments with different transformation temperature ranges. Although iatrogenic complications, such as ledging, zipping, transportation, and file separations heavily depend on operator experience and technique (Shen et al., 2009), it is believed that this study will help some clinicians to learn more about the properties of NiTi in clinical situations.

CHAPTER 2

REVIEW OF THE LITERATURE

The main objective of root canal therapy is to prevent or treat apical periodontitis (Orstavik et al., 1998). One of the key elements of successful root canal therapy is chemomechanical debridement to remove microorganisms and its substrate, and to create a shape acceptable for dense and permanent root canal filling (Schilder, 1974).

In 1965, Kakehashi et al. proposed that the main etiology of apical periodontitis is bacteria. According to their experiment, pulp tissue of germ free rats remained vital while pulp tissue of conventional rats became necrotic and infected, causing apical periodontitis. This was later supported by Sundqvist (1976) who compared human necrotic traumatized teeth with and without apical periodontitis and found that bacteria culture was present only in teeth with apical periodontitis. In addition, Moller et al. (1981) in their study on monkeys found that sterile necrotic pulps did not develop apical periodontitis while infected necrotic pulps had periapical inflammation.

Proper mechanical instrumentation facilitates removal of microorganisms and its substrate and creates a space for irrigant penetration (Schilder, 1974). Mechanical instrumentation alone was shown to reduce bacterial load by 100 to 1000 fold (Bystrom & Sundqvist et al., 1981), but studies have shown that complete canal instrumentation cannot be achieved; according to a micro CT study, 35% of the canal walls remain untouched with current instrumentation techniques (Paque et al., 2010, Rodig et al., 2002). Thus, chemomechanical instrumentation with the use of a disinfecting irrigant is essential for greater bacterial reduction (McGurkin-Smith et al., 2005).

Irrigation has mechanical, chemical, and biological objectives. The mechanical and chemical objectives are to (1) flush out debris, (2) lubricate the canal, (3) dissolve organic and inorganic tissue, and (4) prevent or dissolve a smear layer during instrumentation. The biological objectives are to (1) have an antimicrobial effect against anaerobic and facultative microorganisms in both planktonic and biofilm state, (2) inactivate endotoxin, (3) have non-toxicity when in contact with periodontal tissues, and (4) have little or no potential to cause anaphylactic reaction (Basrani et al., 2012).

One of the most frequently used disinfecting irrigants that achieves many of the above objectives is sodium hypochlorite (NaOCl). NaOCl dissolves proteins and has an antimicrobial effect against microorganisms due to its high pH and its ability to form chloramine (Basrani et al., 2012). Clegg et al. (2006) found that 6% NaOCl to be the only agent capable of both physically removing biofilm and killing bacteria. The study also showed that a lower percentage of NaOCl was equally effective if continuously replenished or given more time to take full effect. Moreover, NaOCl is an effective organic tissue dissolvant as 2.6% of NaOCl solution at 37°C was an equally effective collagen-dissolving solution as 5.2% NaOCl at either 21°C or 37°C (Cunningham and Balekjian et al., 1980).

Another popular irrigant that aids in removing the bacteria is Ethylenediaminetetraacetic acid (EDTA). Although lacking an antimicrobial effect, EDTA effectively removes the smear layer by chelating the inorganic component of dentin and aids disinfecting irrigants like NaOCl to take effect in deeper layers of infected dentin (Haapasalo et al., 2005). Studies have shown that combined use of NaOCl and EDTA resulted in more efficient elimination of bacteria compared to NaOCl without EDTA (Bystrom & Sundqvist et al., 1985, Baumgartner et al., 2007).

There has been debate regarding the appropriate size of apical preparations for successful root canal therapy. According to Salzgeber and Brilliant (1977), the apical preparation should be above size 35 for irrigant to penetrate the apical third of the canal. Abou-Rass (1982) showed that a 30 gauge needle was able to be placed in the apical third of the canal when the apex was prepared to size 30. This was important because irrigant reached the working length only when the side-vented irrigation needle was placed 1 mm from the working length (Boutsioukis et al., 2010). However, some authors claim that there is no significant difference in apical debris removal and intracanal bacterial reduction with or without apical size enlargement if there was suitable coronal taper and shape (Albrecht et al., 2004, Coldero et al., 2002).

Despite the debate on apical size, the general consensus is that cleaning and shaping of the canals to a continuous taper from access cavity to the apical foramen while maintaining the original canal path and apical foramen in its original position are important aspects of successful chemomechanical debridement (Schilder, 1974, Peters et al., 2011).

However, the difficulty arises as the root canal morphology is more complex than portrayed on the 2 dimensional radiograph; unlike curvatures in the mesiodistal plane, curvatures in the faciolingual plane are not apparent on clinical radiographs (Cunningham et al., 1992). In addition, numerous isthmuses, fins, and webs exist between canals as well as variable canal configurations (Hess, 1921). Such a complex and curved nature of canal anatomy may lead to an incidence of iatrogenic complications like ledging, zipping, and canal transportation especially if stiff stainless steel files are used entirely during the canal debridement. Eldeeb et al. (1985) reported that stainless file size 25 or larger has increased risk of zipping and transportation. Other studies have also shown remarkably decreased torsional properties and flexibility on bending of stainless steel files size 35 and larger (Craig et al., 1968).

Factors that attribute to apical blockage, ledge formation, and transportation include instrumentation technique, canal location, tooth type, and canal curvature (Jafarzadeh et al., 2007). Among those factors, canal curvature seems to be the most significant variable affecting the incidence of ledge formation (Kapalas et al., 2000, Greene et al., 1990), which is an artificial irregularity on the root canal surface that impedes the apical advancement of instruments to the otherwise patent canal (AAE Glossary, 2015). Canal transportation may result as the outer wall structure of the canal is removed due to the tendency of stiff stainless files to restore their original linear shape during canal preparation (AAE Glossary, 2015, Jafarzadeh et al., 2007).

Inability to maintain canal anatomy prevents effective bacterial reduction and seal in uninstrumented areas (Jafarzadeh et al., 2007). A systematic review of the literature by Ng et al.

(2007) concluded the 4 significant factors that affect the endodontic outcome as absence of pretreatment apical radiolucency, root filling within 2 mm from the radiographic apex, root filling with no voids, and satisfactory coronal restoration. Chugal et al. (2003) also noted that absence of chronic apical periodontitis, working length maintenance, root canal filling length, and root canal filling density were the main outcome predictors. The study found that teeth with necrotic pulp had the best outcome when the root fillings are 0.55 mm short of the radiographic apex and had no voids, and that a 1 mm loss in working length increased the chance of treatment failure by 14%.

While the search for ideal instruments has continued, it was not until 1988 when the introduction of Nickel-Titanium (NiTi) alloy as an endodontic instrument by Walia et al. (1988) led to a revolution in canal instrumentation. NiTi alloy was first described by W.F. Buehler et al. (1968) in the early 1960s and has been used as orthodontic wires. Later, Walia et al. (1988) reported that size 15 files fabricated from Nickel-Titanium orthodontic alloy (Nitinol) was 2-3 times more flexible in bending and had superior torsional properties than the stainless steel files of the same size and file design. Superior mechanical properties of NiTi alloy showed promise for the instrumentation of the complex root canal system (Schafer et al., 2003).

NiTi alloy can exist as three crystal phases; austenite, martensite, and intermediate R phase (Shen et al., 2013). At high temperature, the alloy exists as the austenite phase, which is a stable, body-centered cubic lattice. When the alloy is cooled through a transformation temperature range, it transforms to a twinned martensitic phase, which is a closely packed hexagonal lattice. Although the change from austenite to twinned martensite is not macroscopically detectable, twinned martensite is more ductile than austenite because it can be easily deformed to detwinned martensite through a single orientation (Thompson, 2000). Unique phase transitions between austenite and martensite renders NiTi its shape memory and superelasticity. Shape memory comes from the fact that NiTi can exist as 2 temperature-dependent crystal structures- monoclinic B19’ for martensite at lower temperature and B2 cubic crystal structure of austenite at high temperature. When NiTi is heated, it transforms from martensite to austenite; the temperature at the start of the austenite transition is called austenite start temperature (As), and temperature at the end of transformation is called austenite finish temperature (Af), and vice versa for martensite start and finish temperatures upon cooling (Buehler et al., 1968, Thompson, 2000). In addition, superelasticity happens when austenite transforms to stress-induced martensite during loading and spontaneously recovers back to austenite when unloaded within a specific temperature range above Af (Kuhn & Jordan, 2002, Duerig et al., 2015). In this state, the application of stress does not result in proportional strain as seen in other metals because superelastic properties of NiTi allows its deformations to be elastic and reversible up to 8% strain compared to a maximum strain of 1-2% with stainless steel (Shen et al., 2013, Thompson, 2000).

NiTi's superior flexibility allows it to be used as an engine-driven rotary file. In 1992, John McSpadden introduced the first rotary 0.02 taper NiTi files into the market. Subsequently, Ben Johnson developed larger taper 0.04 and 0.06 ProFile instruments and orifice openers to compensate for frequent file separation of 0.02 files as well as for improving instrumentation efficiency compared to hand filing with stainless steel hand files (Haapasalo et al., 2013). Studies have supported the use of NiTi files as being more efficient and more centered in the canal compared to stainless steel hand files (Kuhn et al., 1997, Esposito et al., 1995).

Among the first generation of NiTi files is ProFile Series 29 (Tulsa Dental Products, Tulsa, OK, USA), which is characterized by a constant 29.17 % increase of dimension at d_1 between every successive file compared to standard ISO-sized instruments which have variable percent increase at d_1; for example, at the beginning of the series from ISO size 10 to 25, the incremental changes at d_1 is too large. The disadvantage of ProFile Series 29 was that, even though the progression to the next larger instrument was easy in smaller sizes, it was more difficult to progress in the larger sizes especially during instrumentation of curved canals (Schafer et al., 1999).

Then, came second generation files, including K3 (Sybron Endo, Orange, CA), Endosequence (Brasseler, Savannah, GA, USA), and ProTaper (Dentsply Tulsa). Unique features of K3 include a slightly positive rake angle for better cutting efficiency, wide radial lands, peripheral blade relief for reduced friction, and variable pitch and core diameter which makes it stronger close to its apical tip (Haapasalo et al., 2013). However, Chow et al. (2005) examined both K3 and Profile file systems and concluded that all tested K3 files were determined to have negative rake angles and that no studies have evaluated cutting efficiency solely on the criteria of rake angles. Further research is needed to support any benefit of files of a positive or negative rake angle.

Despite the advantages of NiTi instruments, the possibility of file separation, ledge formation and transportation still exist. One of the common iatrogenic errors is file separation, which happens due to torsional failure and cyclic fatigue (Shen et al., 2009). Torsional fracture can happen when the apical portion of the instrument is bound in the canal while the remaining file continues to rotate. Cyclic fatigue is caused by repeated compressive and tensile stress on a rotating file in a curved canal, usually at the maximum point of flexure (Kramkowski et al., 2009). Overall, the incidence of file separation happens about 3-5% of the time (Spilli et al., 2005, Shen et al., 2009, Alapati et al., 2005), and there is a slightly higher prevalence of torsional failure (56%) compared with cyclic fatigue (44%) (Sattapan et al., 2000). The separation of the instrument alone does not affect the endodontic outcome, rather it depends on the location and time of the fracture and whether there is the presence of a pre-operative periapical lesion (Crump and Natkin et al., 1970, Spilli et al., 2005). Meta analysis by Panitvisai et al. (2010) showed that the overall prognosis of the root canal therapy with a retained instrument fragment was 91%, which was not significantly different from the outcome without a retained instrument.

Although file separations and ledge formations heavily depend on operator experience and technique (Shen et al., 2009, Jafarzadeh et al., 2007), numerous attempts have also been made by manufacturers to improve NiTi’s properties to reduce iatrogenic errors. One of the methods proposed is surface finishing via electropolishing. The concept behind it was that polishing or removal of the surface defects like pitting and grooves, which can act as stress concentrators favorable to crack initiation and fracture, would lead to improved resistance to torsional stress and cyclic fatigue (Barbosa et al., 2008). Some authors observed a more regular surface and higher resistance to torsional stress and flexural fatigue after electropolishing (Silva et al., 2011, Anderson et al., 2007). However, other studies have shown that elelctropolishing did not offer significant advantages of microfracture inhibition, cyclic fatigue, cutting efficiency, and torsional resistance (Cheung et al., 2007, Herold et al., 2007, Barbosa et al., 2008, Bui et al., 2008).

In addition, changes in file design, taper and cross-sectional shape can affect the mechanical properties of the file (Camps et al., 1995, Kazemi et al., 2000, Schafer et al., 2003). Cross-sectional geometry, especially, has been shown to be one of the major factors to affect torsion and bending properties (Camps et al., 1995, Xu et al., 2006). Baek et al. (2011) reported that torsional stiffness was increased by cross-sectional geometry, increased cross-sectional area, and reduced pitch (more threads) rather than a difference in inner core area using finite element analysis. The authors noted that a cross-sectional geometry of a rectangle had better torsional properties than a triangle with the same cross-sectional area and larger inner core area. A higher bending moment (less flexibility) was observed for files with a rectangular cross-sectional design than a triangular cross-sectional design, and for files with increased cross-sectional area than smaller cross-sectional area (Camps et al., 1995, Schafer et al., 2003). Also, more residual stress and deformation, which could lead to increased risk of fracture, was observed during simulated shaping of the curved canals for rectangle-based cross-sectional designs compared to triangle-based files (Kim et al., 2009). On the contrary, according to Hayashi et al. (2007), rectangular files were shown to be more flexible than triangular files.

The properties of NiTi are very sensitive and can be affected by chemical composition, manufacturing processes, and heat treatment (Miyazaki et al., 1982, Thompson, 2000).

Thermomechanical processing at about 400°C, especially, was reported to be effective at reducing work hardening, optimizing microstructure, and transformation behavior of NiTi (Kuhn & Jordan et al., 2002). Several recent studies examined the effect of heat treatment on the transformation behavior of the alloy via differential scanning calorimetry (DSC) and found that heat treatment increased the transformation temperature; DSC measures martensitic and reverse transformation temperatures and associated phase transformations between austenite and intermediate R phase and martensite under controlled cooling and heating. The elastic modulus of martensite is lower than austenite, and R phase has an even lower modulus than martensite. Using cantilever bending tests, studies have shown that NiTi instruments with a higher Af have better flexibility because they exist as either a mixture of austenite and R phase or martensite in the oral environment compared with files with a lower Af that exist as austenite (Yahata et al., 2009, Miyai et al., 2006, Hayashi et al., 2007, Ebihara et al., 2011).

Proprietary heat treatment can produce instruments with significantly different Af temperatures and thus improved mechanical properties (Zhou et al., 2012). In 2008, Johnson et al. showed in their experiment that ProFile 25/.04 rotary files manufactured via M-wire technology (Dentsply Tulsa Dental Specialties) had markedly improved cyclic fatigue of up to 390% and equivalent torsional resistance compared with the same instrument design produced from Nitinol SE508 stock. Through this proprietary method of drawing the raw wire under specific tension and thermal treatment at various temperatures, the M-wire alloy includes some portions of both martensite and R-phase while maintaining a pseudoelastic state (Alapati et al., 2009). Soon after, ProFile GTX was introduced as the first commercially available endodontic rotary file using the M-wire technology (Shen et al., 2013). The next generation of M-wire instruments were ProFile Vortex (Dentsply Tulsa Dental Specialties), which showed superior cyclic fatigue and flexibility over conventional NiTi and stainless steel alloy (Johnson et al., 2008, Gao et al., 2012). This contrasted with other studies that showed that M-wire did not perform better than conventional superelastic NiTi (SE-wire) (Gambarini et al., 2008, Al-Sudani et al., 2012).

Later, Vortex Blue (Dentsply Tulsa Dental Specialties) was brought into the market. ProFile Vortex and Vortex Blue have an identical geometric design and differ only in manufacturing process, which results in the blue color of the visible titanium oxide layer. The hardness of the titanium oxide may compensate for the loss of hardness of ProFile Vortex while improving cutting efficiency and wear resistance. Vortex Blue showed a 40% longer fatigue life than M-wire, while M-wire showed 250% better cyclic fatigue than SE-wire. In addition, Vortex Blue was about 13% more flexible than ProFile Vortex, while exhibiting 20% lower maximum torque value (Gao et al., 2012). Plotino et al. (2014) supported this finding as Vortex Blue showed a significantly increased number of cycles to failure when compared with the same size of ProFile Vortex except for the size 15/.04.

Soon after the introduction of the ProFile GTX series, Gambarini et al. (2008) proposed a new manufacturing method of twisting NiTi alloy to produce more flexible and fatigue-resistant NiTi instruments. Prototype Twisted Files (Sybron Endo, Orange, CA) were manufactured by the combination of a special R-phase heat treatment and twisting of metal wire (Hou et al., 2011). Unlike SE wire that cannot maintain its permanent deformation of spiral configuration, R phase wire can be twisted because the transformation strain of R phase is less than one tenth of martensite transformation (Wu et al., 1990, Otsuka et al., 1998). Studies have shown that Twisted Files were significantly more flexible than conventional NiTi files of the same taper and tip size (Gambarini et al., 2008, Hou et al., 2011). An example of recent R-phase technology is K3XF (Sybron Endo, Orange, CA), which has the identical design as K3 but differs in manufacturing process. Studies have shown that K3XF has better fatigue resistance than K3 while maintaining comparable torsional properties as conventional NiTi (Fernandes et al, 2015, Lopes et al., 2013, Shen et al., 2015).

In 2010, HyFlex CM (Coltène/Whaledent Inc., Cuyahoga Falls, OH) files were developed via controlled memory (CM) wire technology, a special heat treatment to render files extremely flexible (Shen et al., 2013). According to the manufacturer, CM wire technology enables the HyFlex files to stay bent without rebounding to its original shape. In addition, HyFlex CM files exhibit lower percent by weight nickel (52 Ni wt%) compared to most 54-57 Ni wt% files on the market. However, the potential role of different percentage in nickel remains uncertain because this compositional deviance comes from raw material variations during manufacturing and thermal processing (Zinelis et al., 2010, Testareli et al., 2011). HyFlex CM files display a mixture of martensite, R-phase, and a small amount of austenite at room and oral temperature, which attributes to superior flexibility and cyclic fatigue resistance compared to conventional NiTi (Testarelli et al., 2011, Zhou et al., 2012). Compared to M-wire, HyFlex has better flexibility, less torque resistance, and higher angles of rotation before fracture. High angle of rotation before fracture may be clinically beneficial because it may indicate imminent fracture by showing visible signs of plastic deformation/unwinding (Ninan & Berzins, 2013).

Of clinical significance, and where a gap in the literature exists, is examining the bending properties of different nickel-titanium rotary files at intracanal temperatures. Intracanal temperature ranges between 31-35°C (Cunningham and Balekjian et al., 1980, Hemptinne et al., 2015); while cantilever-bending tests were done at 37°C (Miyai et al., 2006, Hayata et al., 2007, Yahata et al., 2009, Ebihara et al., 2011), bending tests following the ISO 3630-1 guideline using a torsiometer have been conducted at room temperature (23±2°C) or appear in the literature without specifying the testing temperature.

Studies showed that altered transformation behavior of NiTi alloy at 37°C affected cyclic fatigue resistance for all tested files to a different degree. Overall, there was a decrease in number of cycles to fracture (NCF) but heat-treated files with higher Af temperatures were not as significantly affected as the conventional NiTi files (Vasconcelos et al., 2016, Plotino et al., 2017, Dosanjh et al., 2017). The presence of intracanal irrigant like NaOCl during canal instrumentation is favored by some authors because the irrigant can absorb heat generated by the device, lower the intracanal temperature, improve cutting efficiency by removing intracanal debris, and result in an increased number of cycles to failure (NCF) (Mousavi et al., 2012, Vasconcelos et al., 2016). This is contrasted by some studies that claim that longer exposure to NaOCl may reduce the number of rotations to failure due to corrosion of NiTi (Mize et al., 1998, Peters et al., 2007). However, NiTi alloy has better corrosion resistance compared to stainless steel alloy, and immersion of NiTi in NaOCl has a negligible effect on its mechanical properties (Barbosa et al., 2007, Huang et al., 2017). Further, Haapasalo et al. (2012) evaluated the effect of environmental conditions on fatigue behavior of controlled memory NiTi wire instruments and found that the type of NiTi alloy - CM files versus SE files - influences cyclic fatigue resistance in different environments but also CM files have longer fatigue life in liquid media than in air.

The scope of this study includes six commercially available NiTi rotary instruments with different manufacturing processes, thus having different austenite finish temperatures (Af). The Af of each file was obtained from internal experiment. The hypothesis of this research was that the bending properties of the files will be temperature dependent.

CHAPTER 3

MATERIALS AND METHODS

Six commercially available NiTi files were selected for this experiment. The tested files included K3 40/.04 (Sybron Endo, Orange, CA), ProFile Series 29 Green Size 6 (Dentsply Tulsa Dental Specialties), K3XF 40/.04 (Sybron Endo, Orange, CA), Vortex Blue 40/.04 (Dentsply Tulsa Dental Specialties), ProFile Vortex 40/.04 (Dentsply Tulsa Dental Specialties), and HyFlex CM™ 40/.04 (Coltène/Whaledent Inc., Cuyahoga Falls, OH). The Austenite finish temperatures of the files were 9.6 ± 0.5, 17.6 ± 0.6, 24.9 ± 1.1, 35.4 ± 1.2, 45.7 ± 0.9, and 60.3 ± 3.1, respectively (data obtained internally). The bending properties of the files were measured using a torsiometer (Figure 1; Sabri Dental Enterprises, Inc. Downers Grove, IL) in accordance with ISO 3630-1 guidelines.

For the flexibility test, the tip of each file (3 mm) was placed in the jaws of the chuck perpendicular to the axis of the motor (Figure 2). The catch pin on the motor shaft was mounted and rotated in the clockwise direction until the catch pin was lightly touching the file. The bending moment display was reset to zero, then the motor was activated to rotate 45 degrees and stopped. Finally, the measured peak bending moment was recorded in g·cm.

Twelve of each file type were grouped into three groups based on temperatures. Each temperature group had a total of 72 files. Group 1 measured the bending moment at 9±2°C, group 2 at 23±2°C, and group 3 at 35±2°C.

For group 1, each file was placed in the jaws of the chuck and the catch pin was rotated to touch the file without applying any force. Then, the files in the chuck were stored in a refrigerator for 4 minutes at 9±2°C. The file in the chuck was quickly put back to its position within 10 seconds, and the motor was activated to measure the bending moment of the file when positioned at 45 degrees.

For group 2, the bending moment was measured at 23±2°C, room temperature.

For group 3, a heater (Everstar HPV-25 ceramic heater; Home Depot USA, Inc., Atlanta, GA, USA) was utilized to keep the temperature near the torsiometer at 35±2°C, then the bending moment was measured.

All data was statistically analyzed by ANOVA and *post hoc* HSD with SPSS (SPSS Inc., Chicago, IL) and statistical significance was set at $P<0.05$.

Figure 1. Torsiometer (Sabri Dental Enterprises, Inc. Downers Grove IL)

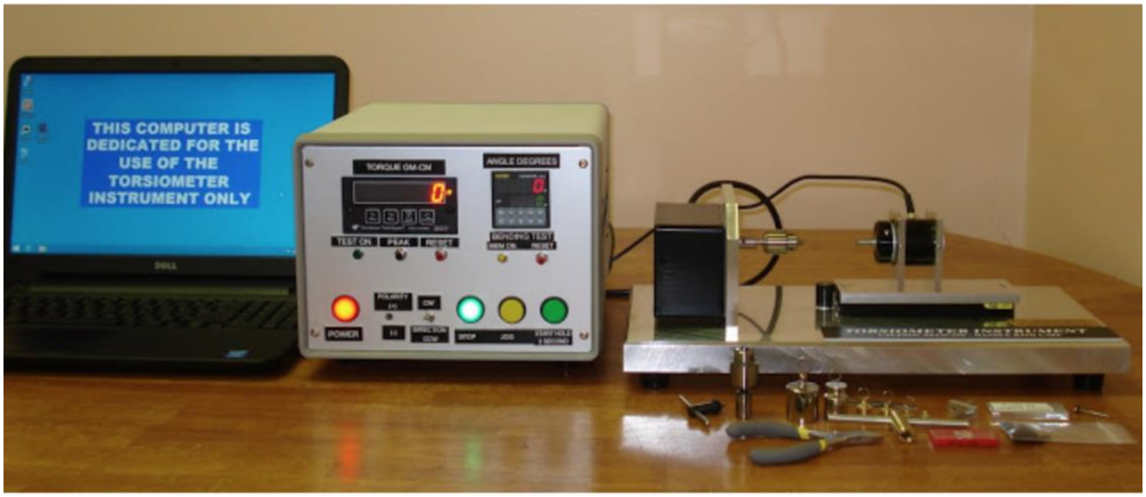
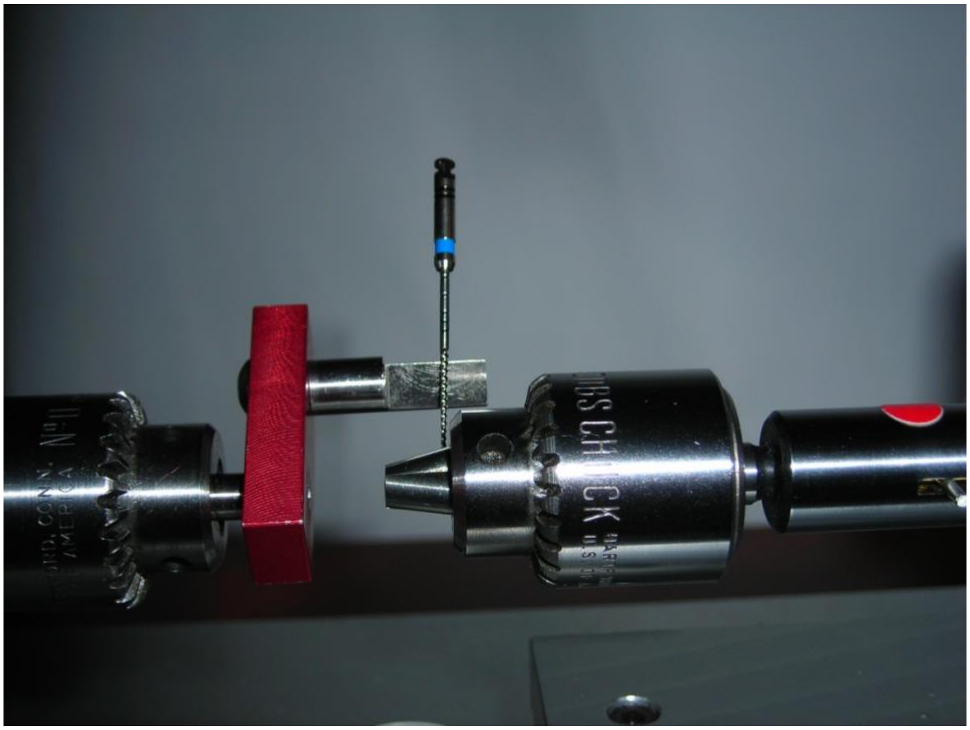


Figure 2. NiTi file set up for bending test in a torsiometer



CHAPTER 4

RESULTS

Table 1 shows the mean bending moment (g·cm) of all the tested files at 9°C, 23°C, and 35°C. For all temperatures, there was a general tendency that the lower the Af, the lower the flexibility of the files. Table 1 shows that the most flexible file was HyFlex CM and the least flexible file was K3. In addition, the rise in temperature resulted in decreased flexibility of the rotary files.

Table 1. Mean (standard deviation) of bending moment (g·cm) of NiTi rotary files

NiTi rotary file brands	**Temperature (°C)**		
	9°C	**23°C**	**35°C**
K3	132 (14)	148 (18)	171 (14)
K3XF	77 (5)	96 (10)	117 (12)
ProFile Vortex	77 (11)	90 (16)	109 (12)
Vortex Blue	78 (7)	97 (12)	118 (17)
HyFlex CM	29 (4)	41 (4)	54 (5)
ProFile Series 29	125 (14)	142 (9)	158 (21)

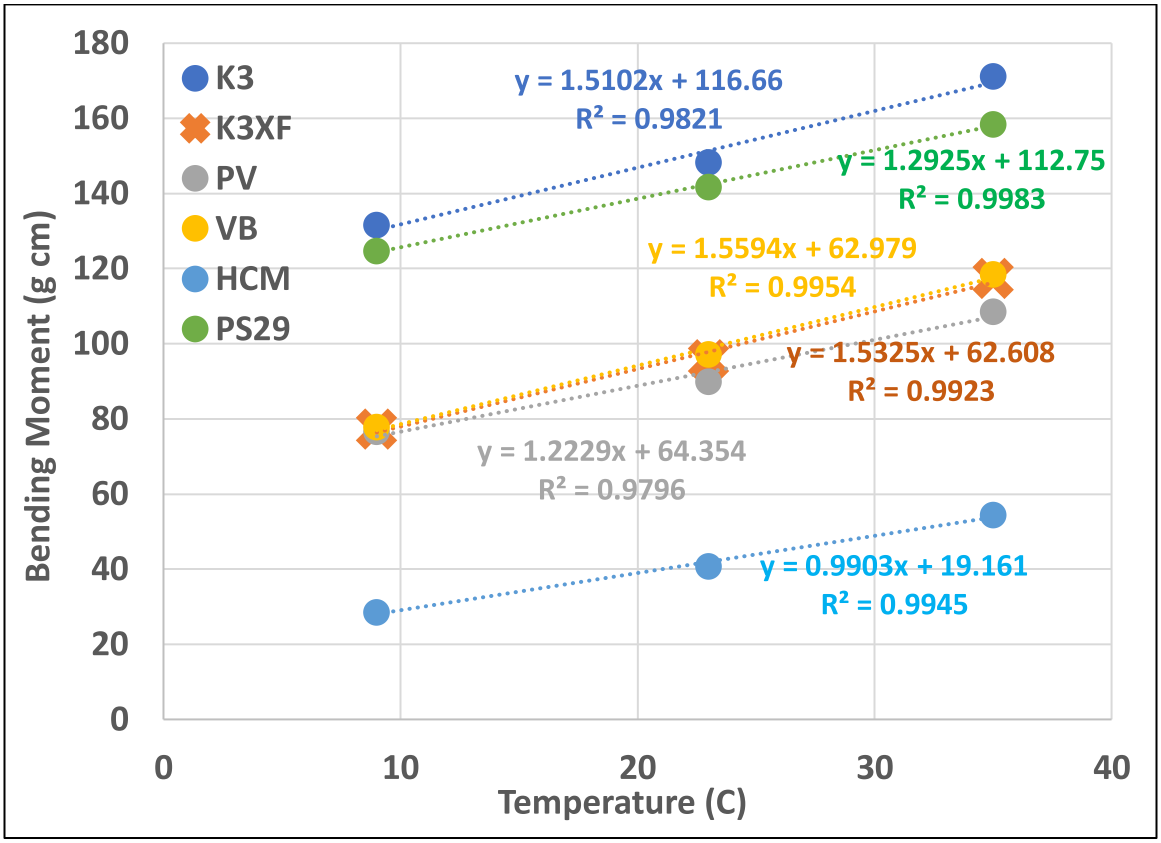
Table 2 shows that there was a significant difference ($p<0.001$) in flexibility between brands and between temperatures. There was no significant interaction ($p>0.05$) between brand and temperature.

Table 2. 2-Way ANOVA tests between-subjects effects
dependent variable: bending moment (g·cm)

Source	**Type III Sum of Squares**	**df**	**Mean Square**	**F**	**Sig**
Brand	278610.986	5	55722.197	365.788	.000
Temp	44904.861	2	22452.431	147.389	.000
Brand*Temp	1091.861	10	109.186	.717	.708

Figure 3 shows again that, for all the file brands, an increase in temperature resulted in decreased flexibility, confirming that there is no significant interaction between the file brands and the temperatures. High R^2 values (0.97-0.99) confirmed that stiffness increases linearly with temperature.

Figure 3. The relationship of the bending moment (g·cm) of the tested files in accordance with the rise in temperature (°C)



According to Table 3, looking at the files as a whole, the files were significantly stiffer as the temperatures increased from 9 to 23 to 35°C. In addition, Table 4 shows that, for all temperatures, K3 had the highest bending moment (stiffer) followed by ProFile Series 29, Vortex Blue, K3XF, ProFile Vortex, HyFlex CM.

Table 3. Flexibiliy of NiTi files in relation to temperatures (°C)
dependent variable: bending moment (g·cm)

Temp (°C)	Mean	Std. Error	95% Confidence Interval	
			Lower Bound	Upper Bound
9.00	86.111	1.455	83.243	88.980
23.00	102.292	1.455	99.423	105.160
35.00	121.389	1.455	118.520	124.257

Table 4. Flexibility of NiTi files in relation to brands
dependent variable: bending moment (g·cm)

Brand	Mean	Std. Error	95% Confidence Interval	
			Lower Bound	Upper Bound
K3	150.389	2.057	146.332	154.445
K3XF	96.833	2.057	92.777	100.890
PV	91.667	2.057	87.610	95.723
Vortex Blue	97.806	2.057	93.749	101.862
HyFlex CM	41.278	2.057	37.221	45.334
PS 29	141.611	2.057	137.555	145.668

According to Table 5, HyFlex CM was significantly ($p<0.05$) more flexible than all other files. K3XF and ProFile Vortex and Vortex Blue were not significantly different ($p>0.05$) from each other and collectively were significantly ($p<0.05$) more flexible than ProFile Series 29. ProFile Series 29 was significantly ($p<0.05$) more flexible than K3.

Table 5. Homogeneous subsets of PostHoc Tukey HSD for brands dependent variable: bending moment (g·cm)					
Brand	**N**	**Subset**			
		1	**2**	**3**	**4**
HyFlex CM	36	41.2778			
PV	36		91.6667		
K3XF	36		96.8333		
Vortex Blue	36		97.8056		
PS 29	36			141.6111	
K3	36				150.3889
Significance		1.000	.286	1.000	1.000

According to Table 6, temperatures produced significantly different bending moments for all the file brands. The files were significantly ($p<0.05$) more flexible at 9°C, followed by at 23°C, and then at 35°C.

Table 6. Homogeneous subset for PostHoc Tukey HSD for temp (°C) dependent variable: bending moment (g·cm)				
Temp (°C)	**N**	**Subset**		
		1	**2**	**3**
9.00	72	86.1111		
23.00	72		102.2917	
35.00	72			121.3889
Significance		1.000	1.000	1.000

CHAPTER 5

DISCUSSION

NiTi files undergo constant development, leading to new generations of NiTi files with different properties. Therefore, it is important for clinicians to understand the characteristics of these new and conventional NiTi instruments before use.

The current study aimed to test how different temperatures affect the bending properties of different NiTi files following the guidelines of ISO 3630-1. The mechanical properties of the files will become more clinically relevant when measured at intracanal temperature because intracanal temperatures are 31-35°C (Cunningham and Balekjian et al., 1980, Hemptinne et al., 2015), the heated or cooled intracanal irrigation solutions will return to equilibrium of body temperature in 1 minute (Hemptinne et al., 2015), and rotary instruments just after removal from a root canal have surface temperatures of 30.8°C-32.5°C (Vasconcelos et al., 2016).

Previous studies showed that altered transformation behavior of NiTi alloy at 37°C reduced fatigue resistance of the files (Plotino et al., 2017, Dosanjh et al., 2017, Vasconcelos et al., 2016). While cantilever-bending tests were done at 37°C (Miyai, et al., 2006, Ebihara et al., 2011, Hayashi et al., 2007, Yahata et al., 2009), bending tests following ISO 3630-1 guideline using a torsiometer were conducted at room temperature (23±2°C) or without specifying the testing temperature (Gao et al., 2012, Testarelli et al., 2011, Gambarini et al., 2008). Therefore, the current study aimed to test how different temperatures (including intracanal temperature) affect the bending properties of different NiTi files.

In the present study, six commercially available NiTi files were selected according to the range of austenite finish temperatures (Af) (internal data). The Af of each instrument listed from lowest to highest are as follows: K3: 9.6°C, ProFile Series 29: 17.6°C, K3XF: 24.9°C, Vortex Blue: 35.4°C, ProFile Vortex: 45.7°C, HyFlex CM: 60.3°C. K3 and ProFile Series 29 have a lower Af than room and body temperature, meaning that they will exist as austenite at both temperatures. K3XF has an Af higher than room temperature but lower than body temperature, thus having a mixture of martensite (or R phase) and austenite at room temperature but existing as austenite clinically. Vortex Blue has an Af similar to intracanal temperature, and it will contain a mixture of martensite and austenite clinically. ProFile Vortex and HyFlex CM with an Af higher than 35°C will present as more martensite clinically.

Martensite and R phase have a lower Elastic modulus than austenite, thus the higher percentage of martensite structure, the more flexible the alloy becomes (Thompson, 2000). Moreover, the existence of martensite has damping properties, having more resistance to crack propagation and fatigue (Shen et al., 2013). Further, a study by Braga et al. (2014) offers the explanation that of the files tested, files with a hybrid austenite-plus-martensite microstructure like that of Hyflex, Profile Vortex, and Typhoon, showed favorable fatigue resistance by comparison with files of fully austenitic microstructure because of the significant number of interfaces. Interfaces have been seen to create secondary cracks which can exhaust the energy needed for crack propagation.

In the present study, for all temperatures, there was a general tendency that the lower the Af, the lower the flexibility of the files. The most flexible file was HyFlex CM and the least flexible file was K3. One of the things that contrasted with previous studies (Gao et al., 2012) was that ProFile Vortex was more flexible than Vortex Blue. Since Vortex Blue and ProFile Vortex have the same file design, it calls attention to other factors affecting flexibility. Vortex Blue has a characteristic 'blue color' titanium oxide surface layer, due to the proprietary manufacturing process, which may compensate for the lack in hardness seen in ProFile Vortex, while also improving the wear resistance or cutting efficiency (Gao et al., 2012).

The result of the present study showed that temperature had a significant effect on the flexibility of all the tested files. There was a direct correlation between temperature and flexibility; the stiffness increased with the rise in temperature. This can be explained by the fact that the heating may cause the transition of the NiTi instruments toward the austenite phase, which makes the alloy stiffer compared to the instruments predominantly in the martensite phase (Viana et al., 2010, Thompson, 2000).

However, the fact that the flexibility of K3 and ProFile Series 29 was reduced even though the crystal structure of the instruments were similar (austenite) at room and intracanal temperature, suggests that other factors may play a role in reducing bending properties. Similar findings were reported for cyclic fatigue resistance at different temperatures (Vasconcelos et al., 2016). In addition, Iijima et al. (2002) showed that NiTi orthodontic wires exhibited higher bending loads for specific deflections as the testing temperature increased even if their wires were above their Af temperature. The result of the present study supports that cyclic fatigue life would be extended at lower temperatures because, if the files are more flexible (less force needed to bend the same amount corresponds to less stress), the resultant lower stress would predict a longer fatigue life. Jamleh, et al. (2016) conducted a similar study evaluating the influence of different surrounding temperatures on cyclic fatigue resistance and deflecting load of superelastic NiTi instrument performance. The study found that the mechanical properties of NiTi instruments were negatively affected at high temperatures and would accelerate instrument fracture and create the need for larger load force to deflect the instrument. The study concluded lower temperatures were found to favorably decrease the deflecting load and extend the lifetime of the superelastic NiTi instrument. The present study agrees with these findings and would further add that at lower temperatures the NiTi instrument is mostly composed of martensite phase which is more flexible and more fatigue resistant as an instrument (Santoro et al., 2001) and that if the instrument is more flexible, less force is needed to bend the instrument, then the reduced stress on the instrument would contribute to a longer cyclic fatigue life.

The result of this study confirms that understanding the transformation behavior with respect to temperature of NiTi is important for clinicians. Previous studies mentioned that canal preparation in the presence of an irrigation solution like NaOCl resulted in reduced intracanal temperature, increased the number of cycles to failure (NCF), and improved cutting efficiency

(Mousavi et al., 2012, Vasconcelos et al., 2016). Some studies suggested heating NaOCl in order to enhance its antimicrobial action as well as its tissue dissolving capacity via preheating the irrigant inside the syringe (Sirtes et al., 2005), heating the irrigant inside the canal with a System B plugger (Sybron Endo, Orange, CA) (Woodmansey, 2005), or using ultrasonic agitation (Zeltner et al., 2009). However, cooling NaOCl can be an option during instrumentation of severely curved canals for improved flexibility of the NiTi instruments. The antibacterial properties of NaOCl will not be affected as long as NaOCl is left to take its action because the temperature of the irrigant will return to equilibrium after 1 minute. Moreover, ultrasonic agitation can be added at the final irrigation step for heating NaOCl and for effective cleaning of microorganisms and infected dentin (Huque and Iwaku et al., 1998), while some authors found no significant difference in intracanal bacterial reduction (Beus and Safavi et al.,2012) or periapical healing with or without ultrasonic activation (Liang et al., 2013).

One of the limitations of this experiment was the in vitro nature of the study. The present study was not conducted using extracted natural teeth to capture the variations in root anatomy, nor the extent and frequency of canal curvatures that may exist within a single canal. The most important factor in the control of fatigue resistance is the diameter of the endodontic file at the point of maximum canal curvature (Braga et al., 2014). Lack of data on file behavior within various curved canals at intracanal temperatures means care should be taken in extrapolating the present results for clinical use.

The present study was conducted with the file allowed to run with no irrigant or use of water bath environment. The study by Haapasalo et al. (2012) concluded that the type of NiTi metal alloy within controlled memory NiTi and conventional superelastic NiTi files influenced the cyclic fatigue resistance in different environments and that for CM files (NEYY and TYP of Clinicians Choice Dental), the fatigue life is longer in liquid media compared to air.

Also, how the flexibility of the files would translate to actual iatrogenic events like ledge formation, zipping, transportation should be further investigated. Moreover, the temperature control relied on a crude method of heating the surrounding dry environment of the file and putting the file in refrigerator. A more ideal setting would be a climate controlled facility such as those in libraries, museums, and historical sites.

Although file design and cross-sectional geometry acted as variables (Camps et al., 1995, Kazemi et al., 2000, Schafer et al., 2003), some files possessed the same file design but with a different Af (K3 and K3XF, ProFile Vortex and Vortex Blue). The change in bending properties according to different temperatures of these files showed that a change in transformation behavior can be a significant factor affecting bending properties.

CHAPTER 6

CONCLUSIONS

Within the limitation of this in vitro study, it can be concluded:

1. The flexibility of the files decreased as the temperature rose from 9°C to 23°C to 35°C.
2. Austenite finish (Af) temperature (transformation behavior) of the files is a major factor determining the flexibility.
3. For Flexibility: HyFlex CM> ProFile Vortex, K3XF, Vortex Blue> ProFile Series 29> K3 at all temperatures.

BIBLIOGRAPHY

Abou-Rass, M. (1982). Evaluation and clinical management of previous endodontic therapy. *The Journal of Prosthetic Dentistry,47*(5), 528-534.

Alapati, S. B., Brantley, W. A., Iijima, M. (2009). Metallurgical characterization of a new nickel-titanium wire for rotary endodontic instruments. *Journal of Endontics,*35(11), 1589–93.

Alapati, S., Brantley, W., Svec, T., Powers, J., Nusstein, J., Daehn, G. (2005). SEM Observations of Nickel-Titanium Rotary Endodontic Instruments that Fractured During Clinical Use. *Journal of Endodontics,31*(1), 40-43.

Albrecht, L., Baumgartner, J., Marshall, J. (2004). Evaluation of Apical Debris Removal Using Various Sizes and Tapers of ProFile GT Files. *Journal of Endodontics,30*(6), 425-428.

Al-Sudani, D., Grande, N. M., Plotino, G. (2012). Cyclic fatigue of nickel-titanium rotary instruments in a double (S-shaped) simulated curvature. *Journal of Endodontics,*38(7), 987–9.

American Assocaition of Endodontists: Glossary of Endodontic Terms. 2015: www.aae.org/glossary.

Anderson, M. E., Price, J. W., Parashos, P. (2007). Fracture Resistance of Electropolished Rotary Nickel-Titanium Endodontic Instruments. *Journal of Endodontics,33*(10), 1212-1216.

Baek, S., Lee, C., Versluis, A., Kim, B., Lee, W., & Kim, H. (2011). Comparison of Torsional Stiffness of Nickel-Titanium Rotary Files with Different Geometric Characteristics. *Journal of Endodontics,37*(9), 1283-1286.

Barbosa, F. O., José, Antônio Da Cunha Ponciano Gomes., Araújo, M. C. (2007). Influence of Sodium Hypochlorite on Mechanical Properties of K3 Nickel-Titanium Rotary Instruments. *Journal of Endodontics,33*(8), 982-985.

Barbosa, F. O., José Antônio Da Cunha Ponciano Gomes, Araújo, M. C. (2008). Influence of Electrochemical Polishing on the Mechanical Properties of K3 Nickel-Titanium Rotary Instruments. *Journal of Endodontics,34*(12), 1533-1536.

Basrani, B., Haapasalo, M. (2012). Update on endodontic irrigating solutions. *Endodontic Topics,27*(1), 74-102.

Baumgartner, J. C., Johal, S., Marshall, J, G. (2007). Comparison of the antimicrobial efficacy of 1.3% NaOCl/BioPure MTAD to 5.25% NaOCl/15% EDTA for root canal irrigation. *Journal of Endodontics,33*(2), 186-186.

Beus, C., Safavi, K., Stratton, J., Kaufman, B. (2012). Comparison of the Effect of Two Endodontic Irrigation Protocols on the Elimination of Bacteria from Root Canal System: A Prospective, Randomized Clinical Trial. *Journal of Endodontics*, *38*(11), 1479-1483.

Boutsioukis, C., Lambrianidis, T., Verhaagen, B., Versluis, M., Kastrinakis, E., Wesselink, P. R., Sluis, L. W. (2010). The Effect of Needle-insertion Depth on the Irrigant Flow in the Root Canal: Evaluation Using an Unsteady Computational Fluid Dynamics Model. *Journal of Endodontics*,36(10), 1664-1668.

Braga, Ligia Carolina Moreira., Silva, Ana Cristina Faria., Buono, Vicente Tadeu Lopes., Bahia, Maria Guiomar de Azevedo. (2014). Impact of heat treatments on the fatique resistance of different rotary nickel-titanium instruments. *Journal of Endodontics,*40(9), 1494-1497.

Buehler, W. J., Wang, F. E. (1968). A summary of recent research on the nitinol alloys and their potential application in ocean engineering. *Ocean Engineering,1*(1), 105-120.

Bui, T. B., Mitchell, J. C., Baumgartner, J. C. (2008). Effect of Electropolishing ProFile Nickel–Titanium Rotary Instruments on Cyclic Fatigue Resistance, Torsional Resistance, and Cutting Efficiency. *Journal of Endodontics,34*(2), 190-193.

Byström, A., Sundqvist, G. (1981). Bacteriologic evaluation of the efficacy of mechanical root canal instrumentation in endodontic therapy. *European Journal of Oral Sciences,89*(4), 321-328.

Byström, A., Sunvqvist, G. (1985). The antibacterial action of sodium hypochlorite and EDTA in 60 cases of endodontic therapy. *International Endodontic Journal,18*(1), 35-40.

Camps, J. J., Pertot, W. J., Levallois, B. (1995). Relationship between file size and stiffness of nickel titanium instruments. *Dental Traumatology,11*(6), 270-273.

Cheung, G. S., Shen, Y., Darvell, B. W. (2007). Does Electropolishing Improve the Low-cycle Fatigue Behavior of a Nickel–Titanium Rotary Instrument in Hypochlorite? *Journal of Endodontics,33*(10), 1217-1221.

Chow, D. Y., Stover, S. E., Bahcall, J. K., Jaunberzins, A., Toth, J. M. (2005). An in vitro comparison of the rake angles between K3 and ProFile endodontic file systems. *Journal of Endodontics*,31(3), 180-182.

Chugal, N. M., Clive, J. M., Spångberg, L. S. (2003). Endodontic infection: some biologic and treatment factors associated with outcome. *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology, and Endodontology,96*(1), 81-90.

Clegg, M., Vertucci, F., Walker, C., Belanger, M., Britto, L. (2006). The Effect of Exposure to Irrigant Solutions on Apical Dentin Biofilms In Vitro. *Journal of Endodontics,32*(5), 434-437.

Coldero, L. G., Mchugh, S., Mackenzie, D., Saunders, W. P. (2002). Reduction in intracanal bacteria during root canal preparation with and without apical enlargement. *International Endodontic Journal,35*(5), 437-446.

Craig, R., Mcilwain, E., Peyton, F. (1968). Bending and torsion properties of endodontic instruments. *Oral Surgery, Oral Medicine, Oral Pathology,25*(2), 239-254.

Crump, M. C., Natkin, E. (1970). Relationship of Broken Root Canal Instruments to Endodontic Case Prognosis: A Clinical Investigation. *The Journal of the American Dental Association,80*(6), 1341-1347.

Cunningham, W. T., Balekjian, A. Y. (1980). Effect of temperature on collagen-dissolving ability of sodium hypochlorite endodontic irrigant. *Oral Surgery, Oral Medicine, Oral Pathology,49*(2), 175-177.

Cunningham, C. J., Senia, E. S. (1992). A three-dimensional study of canal curvatures in the mesial roots of mandibular molars. *Journal of Endodontics,18*(6), 294-300.

Dosanjh, A., Paurazas, S., Askar, M. (2017). The Effect of Temperature on Cyclic Fatigue of Nickel-titanium Rotary Endodontic Instruments. *Journal of Endodontics,43*(5), 823-826.

Duerig, T. W., Bhattacharya, K. (2015). The Influence of the R-Phase on the Superelastic Behavior of NiTi. *Shape Memory and Superelasticity,1*(2), 153-161.

Ebihara, A., Yahata, Y., Miyara, K., Nakano, K., Hayashi, Y., Suda, H. (2011). Heat treatment of nickel-titanium rotary endodontic instruments: effects on bending properties and shaping abilities. *International Endodontic Journal,44*(9), 843-849.

Eldeeb, M. E., Boraas, J. C. (1985). The effect of different files on the preparation shape of severely curved canals. *International Endodontic Journal,18*(1), 1-7.

Esposito, P. T., Cunningham, C. J. (1995). A comparison of canal preparation with nickel-titanium and stainless steel instruments. *Journal of Endodontics,21*(4), 173-176.

Fernandes, F. M., Oliveira, J. P., Machado, A. M., Alves, A. R., Schell, N. (2015). Effect of heat treatment on K3, K3XF and MTwo endodontic files. *MATEC Web of Conferences,33*, 03016.

Gambarini, G., Gerosa, R., Luca, M. D., Garala, M., Testarelli, L. (2008). Mechanical properties of a new and improved nickel-titanium alloy for endodontic use: an evaluation of file flexibility. *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology, and Endodontology,105*(6), 798-800.

Gao, Y., Gutmann, J. L., Wilkinson, K., Maxwell, R., Ammon, D. (2012). Evaluation of the Impact of Raw Materials on the Fatigue and Mechanical Properties of ProFile Vortex Rotary Instruments. *Journal of Endodontics,38*(3), 398-401.

Greene, K. J., Krell, K. V. (1990). Clinical factors associated with ledged canals in maxillary and mandibular molars. *Oral Surgery, Oral Medicine, Oral Pathology,70*(4), 490-497.

Haapasalo, M., Endal, U., Zandi, H., Coil, J. M. (2005). Eradication of endodontic infection by instrumentation and irrigation solutions. *Endodontic Topics,10*(1), 77-102.

Haapasalo, M., Shen, Y. (2013). Evolution of nickel-titanium instruments: from past to future. *Endodontic Topics,29*(1), 3-17.

Hayashi, Y., Yoneyama, T., Yahata, Y., Miyai, K., Doi, H., Hanawa, T., Ebihara, A., Suda, H.

(2007). Phase transformation behaviour and bending properties of hybrid nickel-titanium rotary endodontic instruments. *International Endodontic Journal,40*(4), 247-253.

Hemptinne, F. D., Slaus, G., Vandendael, M., Jacquet, W., Moor, R. J., Bottenberg, P. (2015). In Vivo Intracanal Temperature Evolution during Endodontic Treatment after the Injection of Room Temperature or Preheated Sodium Hypochlorite. *Journal of Endodontics,41*(7), 1112-1115.

Herold, K. S., Johnson, B. R., Wenckus, C. S. (2007). A Scanning Electron Microscopy Evaluation of Microfractures, Deformation and Separation in EndoSequence and Profile Nickel-Titanium Rotary Files Using an Extracted Molar Tooth Model. *Journal of Endodontics,33*(6), 712-714.

Hess, W. (1921). Formation of Root-Canals in Human Teeth11"Zur Anatomie der Wurzelkanale des menschliches Gebisses," Schweizerische Vierteljahrsschrift fur Zahnheilkunde, 1917. Translated by Newton G. Thomas, A.B., M.A., D.D.S., and Herman Redlich, Chicago, Illinois. *The Journal of the National Dental Association,8*(9), 704-734.

Hou, X., Yahata, Y., Hayashi, Y., Ebihara, A., Hanawa, T., Suda, H. (2011). Phase transformation behaviour and bending property of twisted nickel-titanium endodontic instruments. *International Endodontic Journal,44*(3), 253-258.

Huang, X., Shen, Y., Wei, X., Haapasalo, M. (2017). Fatigue Resistance of Nickel-titanium Instruments Exposed to High-concentration Hypochlorite. *Journal of Endodontics,43*(11), 1847-1851.

Huque, J., Kota, K. P., Yamaga, M., Iwaku, M., Hoshino, E. (1998). Bacterial eradication from root dentine by ultrasonic irrigation with sodium hypochlorite. *International Endodontic Journal,31*(4), 242-250.

Iijima, M., Ohno, H., Kawashima, I., Endo, K., Mizoguchi, I. (2002). Mechanical behavior at different temperatures and stresses for superelastic nickel-titanium orthodontic wires having different transformation temperatures. *Dental Materials,*18(1), 88-93.

International Organization for Standardization 3630-1. (2008). Dentistry-root canal instruments: Part I - general requirements and test methods. International Organization for Standardization: Geneva, Switzerland.

Jafarzadeh, H., Abbott, P. V. (2007). Ledge Formation: Review of a Great Challenge in Endodontics. *Journal of Endodontics,33*(10), 1155-1162.

Jamleh, A., Yahata, Y., Ebihara, A., Atmeh, A. R., Bakhsh, T., Suda, H. (2016). Performance of NiTi endodontic instruments under different temperatures. *Odontology,*104(3), 324-328.

Johnson, E., Lloyd, A., Kuttler, S., Namerow, K. (2008). Comparison between a Novel Nickel-Titanium Alloy and 508 Nitinol on the Cyclic Fatigue Life of ProFile 25/.04 Rotary Instruments. *Journal of Endodontics,34*(11), 1406-1409.

Kakehashi, S., Stanley, H., Fitzgerald, R. (1965). The effects of surgical exposures of dental pulps in germ-free and conventional laboratory rats. *Oral Surgery, Oral Medicine, Oral*

Pathology,20(3), 340-349.

Kapalas, A., Lambrianidis, T. (2000). Factors associated with root canal ledging during instrumentation. *Dental Traumatology,16*(5), 229-231.

Kuhn, W. G., Carnes, D. L., Clement, D. J., Walker, W. A. (1997). Effect of tip design of nickel-titanium and stainless steel files on root canal preparation. *Journal of Endodontics,23*(12), 735-738.

Kazemi, R. B., Stenman, E., Spångberg, L. S. (2000). A comparison of stainless steel and nickel-titanium H-type instruments of identical design: Torsional and bending tests. *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology, and Endodontology,90*(4), 500-506.

Kim, H. C., Kim, H. J., Lee, C. J., Kim, B. M., Park, J. K., Versluis, A. (2009). Mechanical response of nickel-titanium instruments with different cross-sectional designs during shaping of simulated curved canals. *International Endodontic Journal,42*(7), 593-602.

Kramkowski, T. R., Bahcall, J. (2009). An In Vitro Comparison of Torsional Stress and Cyclic Fatigue Resistance of ProFile GT and ProFile GT Series X Rotary Nickel-Titanium Files. *Journal of Endodontics,35*(3), 404-407.

Kuhn, G., Jordan, L. (2002). Fatigue and Mechanical Properties of Nickel-Titanium Endodontic Instruments. *Journal of Endodontics,28*(10), 716-720.

Liang, Y. H., Jiang, L. M., Jiang, L., Chen, X. B., Liu, Y. Y., Tian, F. C., Bao, X. D., Gao, X. J., Versluis, M., Wu, M. K., Sluis, L. V. (2013). Radiographic Healing after a Root Canal Treatment Performed in Single-rooted Teeth with and without Ultrasonic Activation of the Irrigant: A Randomized Controlled Trial. *Journal of Endodontics, 39*(10), 1218-1225.

Lopes, H. P., Gambarra-Soares, T., Elias, C. N., Siqueira, J. F., Inojosa, I. F., Lopes, W. S., Vieira, V. T. (2013). Comparison of the Mechanical Properties of Rotary Instruments Made of Conventional Nickel-Titanium Wire, M-Wire, or Nickel-Titanium Alloy in R-Phase. *Journal of Endodontics,39*(4), 516-520.

Mcgurkinsmith, R., Trope, M., Caplan, D., Sigurdsson, A. (2005). Reduction of Intracanal Bacteria Using GT Rotary Instrumentation, 5.25% NaOCl, EDTA, and Ca(OH)2. *Journal of Endodontics,31*(5), 359-363.

Miyai, K., Ebihara, A., Hayashi, Y., Doi, H., Suda, H., Yoneyama, T. (2006). Influence of phase transformation on the torsional and bending properties of nickel-titanium rotary endodontic instruments. *International Endodontic Journal,39*(2), 119-126.

Miyazaki, S., Ohmi, Y., Otsuka, K., Suzuki, Y. (1982). Characteristics of deformation and transformation pseudoelasticity in Ti-Ni alloys. *Le Journal de Physique Colloques,43*(C4).

Mize, S. B., Clement, D. J., Carnes, D. L., Pruett, J. P. (1998). Effect of sterilization on cyclic fatigue of rotary nickel-titanium endodontic instruments. *Journal of Endodontics*,24(12), 843-847.

Möller, A. J., Fabricius, L., Dahlén, G., Öhman, A. E., Heyden, G. (1981). Influence on periapical tissues of indigenous oral bacteria and necrotic pulp tissue in monkeys. *European Journal of Oral Sciences,89*(6), 475-484.

Mousavi S. A., Kargar-Dehnavi, V., Mousavi, S. A. (2012). A novel approach to determine the effect of irrigation on temperature and failure of Ni-Ti endodontic rotary files. *Dental Research Journal,*9(3), 281-287.

Ng, Y., Mann, V., Rahbaran, S., Lewsey, J., Gulabivala, K. (2007). Outcome of primary root canal treatment: systematic review of the literature – Part 2. Influence of clinical factors. *International Endodontic Journal,*41(1), 6-31.

Ninan, E., Berzins, D. W. (2013). Torsion and Bending Properties of Shape Memory and Superelastic Nickel-Titanium Rotary Instruments. *Journal of Endodontics,39*(1), 101-104.

Ørstavik, D., Pitt Ford, T. R. (1998). *Essential endodontology: prevention and treatment of apical periodontitis*. Oxford, UK: Blackwell Munksgaard.

Otsuka, K., Wayman, C. M. (1998) Shape Memory Alloys, 1st edn. Cambridge, UK: Cambridge University Press.

Panitvisai, P., Parunnit, P., Sathorn, C., Messer, H. H. (2010). Impact of a Retained Instrument on Treatment Outcome: A Systematic Review and Meta-analysis. *Journal of Endodontics,36*(5), 775-780.

Paqué, F., Balmer, M., Attin, T., Peters, O. A. (2010). Preparation of Oval-shaped Root Canals in Mandibular Molars Using Nickel-Titanium Rotary Instruments: A Micro-computed Tomography Study. *Journal of Endodontics,36*(4), 703-707.

Peters, O. A., Roehlike, J. O., Baumann, M. A. (2007). Effect of Immersion in Sodium Hypochlorite on Torque and Fatigue Resistance of Nickel-Titanium Instruments. *Journal of Endodontics,33*(5), 589-593.

Peters, O. A., & Peters, C. I. (2011). Cleaning and Shaping of the Root Canal System. *Cohens Pathways of the Pulp,*283-348.

Plotino, G., Grande, N. M., Bellido, M. M., Testarelli, L., Gambarini, G. (2017). Influence of Temperature on Cyclic Fatigue Resistance of ProTaper Gold and ProTaper Universal Rotary Files. *Journal of Endodontics,43*(2), 200-202.

Plotino, G., Grande, N. M., Cotti, E., Testarelli, L., Gambarini, G. (2014). Blue Treatment Enhances Cyclic Fatigue Resistance of Vortex Nickel-Titanium Rotary Files. *Journal of Endodontics,40*(9), 1451-1453.

Rodig, T., Hulsmann, M., Muhge, M., Schafers, F. (2002). Quality of preparation of oval distal root canals in mandibular molars using nickel-titanium instruments. *International Endodontic Journal,35*(11), 919-928.

Salzgeber, R. M., Brilliant, J. D. (1977). An in vivo evaluation of the penetration of an irrigating solution in root canals. *Journal of Endodontics,3*(10), 394-398.

Santoro, M., Nicolay, O. F., Cangialosi, T. J. (2001). Pseudoelasticity and thermoelasticity of nickel-titanium alloys: A clinically oriented review. Part I: Temperature transitional ranges. *American Journal of Orthodontics and Dentofacial Orthopedics*,119(6), 587-593.

Sattapan, B., Nervo, G., Palamara, J., Messer, H. (2000). Defects in Rotary Nickel-Titanium Files After Clinical Use. *Journal of Endodontics,26*(3), 161-165.

Schäfer, E., Dzepina, A., Danesh, G. (2003). Bending properties of rotary nickel-titanium instruments. *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology, and Endodontology,96*(6), 757-763.

Schäfer, E., Lau, R. (1999). Comparison of cutting efficiency and instrumentation of curved canals with nickel-titanium and stainless-steel instruments. *Journal of Endodontics,25*(6), 427-430.

Schäfer, E., Dzepina, A., & Danesh, G. (2003). Bending properties of rotary nickel-titanium instruments. *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology, and Endodontology,96*(6), 757-763.

Schilder, H. (1974) Cleaning and shaping the root canal. *Dental Clinics of North America*,18(2), 269-296.

Shen, Y., Zhou, H., Zheng, Y., Peng, B., & Haapasalo, M. (2013). Current Challenges and Concepts of the Thermomechanical Treatment of Nickel-Titanium Instruments. *Journal of Endodontics,39*(2), 163-172.

Shen, Y., Cheung, G. S., Peng, B., & Haapasalo, M. (2009). Defects in Nickel-Titanium Instruments after Clinical Use. Part 2: Fractographic Analysis of Fractured Surface in a Cohort Study. *Journal of Endodontics,35*(1), 133-136.

Shen, Y., Riyahi, A. M., Campbell, L., Zhou, H., Du, T., Wang, Z., Qian, W., Haapasalo, M. (2015). Effect of a Combination of Torsional and Cyclic Fatigue Preloading on the Fracture Behavior of K3 and K3XF Instruments. *Journal of Endodontics,41*(4), 526-530.

Shen, Y., Qian, W., Abtin, H., Gao, Y., Haapasalo, M. (2012). Effect of environment on fatigue failure of controlled memory wire nickel-titanium rotary instruments. *Journal of Endodontics,*38(3), 376-80.

Silva, M. A., José Antônio Da C. Ponciano Gomes, Ormiga, F. (2011). Influence of electrochemical polishing on the mechanical behaviour of nickel-titanium rotary files. *Australian Endodontic Journal,39*(2), 73-77.

Sirtes, G., Waltimo, T., Schaetzle, M., Zehnder, M. (2005). The Effects of Temperature on Sodium Hypochlorite Short-Term Stability, Pulp Dissolution Capacity, and Antimicrobial Efficacy. *Journal of Endodontics,31*(9), 669-671.

Spili, P., Parashos, P., Messer, H. H. (2005). The Impact of Instrument Fracture on Outcome of Endodontic Treatment. *Journal of Endodontics,31*(12), 845-850.

Sundqvist, G. (1976). Bacteriological studies of necrotic dental pulps. *Umae Univ Odontological Dissertations*,7, 1-93.

Testarelli, L., Plotino, G., Al-Sudani, D., Vincenzi, V., Giansiracusa, A., Grande, N. M., Gambarini, G. (2011). Bending Properties of a New Nickel-Titanium Alloy with a Lower Percent by Weight of Nickel. *Journal of Endodontics,37*(9), 1293-1295.

Thompson, S. A. (2000). An overview of nickel-titanium alloys used in dentistry. *International Endodontic Journal,33*(4), 297-310.

Vasconcelos, R. A., Murphy, S., Carvalho, C. A., Govindjee, R. G., Govindjee, S., Peters, O. A. (2016). Evidence for Reduced Fatigue Resistance of Contemporary Rotary Instruments Exposed to Body Temperature. *Journal of Endodontics,42*(5), 782-787.

Viana, A. C., Melo, M. C., Bahia, M. G., & Buono, V. T. (2010). Relationship between flexibility and physical, chemical, and geometric characteristics of rotary nickel-titanium instruments. *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology, and Endodontology,110*(4), 527-533.

Walia, H., Brantley, W. A., & Gerstein, H. (1988). An initial investigation of the bending and torsional properties of nitinol root canal files. *Journal of Endodontics,14*(7), 346-351.

Woodmansey, K. F. (2005). Intracanal heating of sodium hypochlorite solution: an improved endodontic irrigation technique. *Dentistry Today*,24(10),114–116.

Wu, S. K., Lin, H. C., Chou, T. S. (1990) A study of electrical resistivity, internal friction and shear modulus on an aged $Ti_{49}Ni_{51}$ alloy. *Acta Metallurgica et Materialia,*38(1), 95–102.

Xu, X., Eng, M., Zheng, Y., & Eng, D. (2006). Comparative Study of Torsional and Bending Properties for Six Models of Nickel-Titanium Root Canal Instruments with Different Cross-Sections. *Journal of Endodontics,32*(4), 372-375.

Yahata, Y., Yoneyama, T., Hayashi, Y., Ebihara, A., Doi, H., Hanawa, T., & Suda, H. (2009). Effect of heat treatment on transformation temperatures and bending properties of nickel-titanium endodontic instruments. *International Endodontic Journal,42*(7), 621-626.

Zeltner, M., Peters, O. A., Paque, F. (2009). Temperature changes during ultrasonic irrigation with different inserts and modes of activation. *Journal of Endodontics*,35(4), 573–577.

Zhou, H., Shen, Y., Zheng, W., Li, L., Zheng, Y., & Haapasalo, M. (2012). Mechanical Properties of Controlled Memory and Superelastic Nickel-Titanium Wires Used in the Manufacture of Rotary Endodontic Instruments. *Journal of Endodontics,38*(11), 1535-1540.

Zinelis, S., Eliades, T., Eliades, G. (2010). A metallurgical characterization of ten endodontic Ni-Ti instruments: assessing the clinical relevance of shape memory and superelastic properties of Ni-Ti endodontic instruments. *International Endodontic Journal,43*(2), 125-134.