

Influence of an Innovative Anti-Corrosive Solution on Resistance of Endodontic NiTi Rotary Instruments: A Preliminary Study

Mohammed Ali SAGHIRI, Armen ASATOURIAN, Franklin GARCIA GODOY, Nader SHEIBANI

ABSTRACT

Objective: To evaluate the effects of deionised water, blood, phosphate-buffered saline (PBS) and a new anti-corrosive solution based on methoxy propyl amine (MOPA) on the cyclic fatigue resistance of endodontic NiTi rotary instruments under in vitro conditions.

Methods: Forty ProTaper F1 files were provided and divided to four groups (n=10). Samples were first autoclaved and then stored in deionised water, blood, PBS or MOPA for 24 hours. Cyclic fatigue was tested with a custom-made stainless-steel block including artificial canals (curvature angle=30 degree, radius of curvature=5 mm). After immersion in test solutions, samples were rotated 300 rpm until fracture occurred. The number of cycles to failure (NCF) was calculated using recorded fracture time.

Results: Data were analysed by the Kolmogorov–Smirnov, Levene, ANOVA and Scheffe statistical tests. Samples in blood group showed the lowest and samples in MOPA group showed the highest NCF values. Significant difference was observed between groups (p=0.001). NCF value of PBS group was significantly more than the NCF values of samples in blood and deionised water groups (p<0.05).

Conclusion: The tested novel anti-corrosive solution significantly increased the fracture resistance of the endodontic NiTi rotary instruments by reducing the cyclic fatigue. In contrast, blood and deionised water caused more corrosion and resulted in earlier file fracture.

Keywords: anti-corrosion, cyclic fatigue, NCF, NiTi rotary

Please cite this article as: Saghiri MA, Asatourian A, Garcia Godoy F, Sheibani N. Influence of an Innovative Anti-Corrosive Solution on Resistance of Endodontic NiTi Rotary Instruments: A Preliminary Study. Eur Endod J 2018; 3: 55-60

From the Department of Ophthalmology & Visual Sciences and Biomedical Engineering (M.A.S. ✉saghiri@wisc.edu), University of Wisconsin School of Medicine and Public Health, Wisconsin, USA; Department of Angiogenesis and Regenerative Medicine Sector (A.A.), Dr. H. Afsar Lajevardi Research Cluster, Shiraz, Iran; Department of Dentistry (F.G.G.), University of Tennessee Health Science Center, Tennessee, USA; Department of Ophthalmology & Visual Sciences, Cell and Regenerative Biology, and Biomedical Engineering (N.S.), University of Wisconsin School of Medicine and Public Health, Madison, Wisconsin, USA

Received 18 June 2017, revision requested 14 August 2017, last revision received 1 September 2017, accepted 9 September 2017.

Published online: 21 December 2017
DOI 10.5152/eej.2017.17043

HIGHLIGHTS

- Deionized water and blood can negatively influence the structure of NiTi made devices.
- Methoxy propyl amine (MOPA) novel solution is an anti-corrosive agent, which can increase the cyclic *fatigue resistance* and delay separation of NiTi made rotary instruments
- NiTi rotary files in the bleeding root canals might be threatened in the presence of blood. However, the contact time in root canals is much lower than storage time considered in this study.

INTRODUCTION

The advent of NiTi rotary instruments to the field of endodontic was a revolutionary change. These devices are made of pseudo-elastic alloy of nickel and titanium (Nitinol 55) and have an incredible influence on the mechanical preparations of root canals (1). The instrumentation of narrow and curved root canals can be challenging for many clinicians. Walia et al. showed that Nitinol files are at least two to three times more flexible than stainless steel (SS) files (2). The flexibility

can enable the instrument to be used in curved and complex root canals with more facility and lower root canal shape alterations (3).

Many investigators have shown that the unexpected separation of NiTi rotary instruments, apart from higher strength and flexibility, remains a clinical challenge. This undesirable event can occur without any significant deformation seen on the surface of instrument (4). Different causes, such as cyclic fatigue, static and dynamic torsional fatigues, have been attributed to the fracture of these devices (5). Cyclic fatigue is described by the alternating tension and compression cycles, which occur inside the structure of an instrument when it is moving through the maximum curvature of root canal (6). The cyclic fatigue is generally a concern in curved canals, and the many

determinants such as radius and degree of canal curvature, instrument mass, diameter, taper, extended usage and also operator experience could contribute to the occurrence of this fatigue (5, 7-9). Torsional fatigue is considered to be another reason for instrument separation, which can also happen in straight root canals. This fatigue occurs due to instrument lock inside the canal, whereas the engine of hand piece is trying to rotate the shank of the instrument at the same time (7).

Investigations have been made to enhance the cyclic fatigue resistance of endodontic NiTi rotary instruments. Some authors have discussed whether new manufacturing methods can increase the fatigue resistance. Twisted method was introduced as one of the manufacturing methods that can increase the fatigue resistance of end product in comparison to the traditional grinding process (10). Other investigators reported that thermal treatments such as annealing of these instruments can enhance their fatigue resistance (11). Apart from reasons mentioned for the fatigue failure of NiTi instruments, others have suggested that the corrosion mechanisms, as a result of chemomechanical preparation and chemical disinfection or sterilisation, might be an initial factor for fatigue failure. These authors have questioned the effect of pitting or crevice corrosion on the surface of endodontic NiTi rotary instruments (12, 13).

Fretting corrosion occurs on the surface of corrosive substrate when it comes into contact with another surface during an oscillating movement. This issue was first seen for orthopaedic implants such as hip or knee joints, which resulted in corrosion of these implants and reduced the lifetime of these devices (14). There is a passivating oxide film of TiO_2 on the surface of titanium and its alloys, which can be rubbed off or deteriorate due to low shear stresses such as rubbing toward another surface. Fretting corrosion is another reason for disruption of the passive oxide layer (14, 15). In the medical field, some authors have discussed fretting corrosion effect on crevice corrosion of modular hip tapers, which could accelerate the process of crevice corrosion (16). Based on the effect of pitting or crevice corrosion on cyclic fatigue failure of endodontic NiTi rotary instruments, it can be presumed that fretting corrosion is the initial process, which leads to crevice corrosion and finally resulting in cyclic fatigue failure (13).

There have been many types of solutions, which are used to prevent, inhibit and/or minimise corrosion. Corrosion inhibitors are one of the popular solutions that serve this goal (17-20). Generally, the corrosion inhibitors are categorised into two separate categories. First category includes the inhibitors that are coated on the surface of substrates in order to protect the substrate from any contact with oxidative agents, and the second category includes the migrating corrosion inhibitors (MCI), which should be applied when the substrate is under rehabilitation procedures. These inhibitors are able to penetrate into the substrate and remove the oxygen from the tex-

ture of substrate. This category of inhibitors contains basically mixtures of amines or alkoilichilic compounds (21, 22).

A novel anti-corrosive solution was recently patented (US patent 20160024311), which contains methoxy propyl amine (3-methoxypropyl-amine) (MOPA). MOPA is a colourless and clear liquid with ammoniacal odour, which is soluble in water and other organic solvents (23). MOPA has been used as a corrosion-inhibitory solution in steam condensate systems. The anti-corrosive effect of MOPA is due to its amine content (24). The present study aimed to evaluate the effect of different environments including deionised water, blood, PBS and MOPA on the cyclic fatigue resistance of endodontic NiTi rotary instruments. We determined whether MOPA can increase the fatigue resistance due to its anti-corrosive potential under in vitro conditions.

METHODS

A stainless-steel block, 110 mm wide (side-by-side), 100 mm long and 10 mm deep, was made of hardened stainless steel. This block was provided by using a Computer Numerical Control (CNC) device (Fig. 1A). The artificial canals were latched to the length of 16 mm, radius 5 mm and a 30-degree angle. Similar to a previously done study (25), an apparatus was used as a frame for supporting the rotary instrument hand piece for rotating the instruments inside the artificial canals.

Samples Storage

Forty ProTaper F1 files (Maillefer-Dentsply, Baillagues, Switzerland) were selected and autoclaved two times and divided into four groups of 10 each ($n=10$). ProTaper F1 files were firstly rotated at 300 rpm for 30 s in the presence of PBS and then underwent two cycles of sterilisation at 121°C, 15 psi for 15 min. Thereafter, samples were immersed in 10-mL glass vials containing 5 mL of the following solutions for 24 h. G1: Deionised water (DW); G2: blood; G3: PBS and G4: novel anti-corrosive solution (MOPA) (pH=11.1). Blood was collected by a vein puncture needle 25×7 (Vacutainer) in a 5-mL tube with 5% anticoagulant EDTA, percentage by weight.

Cyclic Fatigue Testing

At first, all instruments were inserted into artificial canals to the full length and rotated using a 1:16 reduction Anthogyr hand piece (Sallanches, France) with a torque-controlled electric (speed=300 rpm, torque=70 Nm, according to manufacturer's instructions). NCF has been recorded for each instrument ($NCF=time \times speed$).

SEM Analysis

Samples before and after sterilisation and also after file separation were evaluated by a scanning electron microscope. Machine was operating at 15 kV (Leo. 440i; Oxford Microscopy, Oxford, UK). Data were analysed by Kolmogorov-Smirnov test, Levene test, ANOVA test and Scheffe test using SPSS software version 21 (IBM Corp.; Armonk, NY, USA). The level of significance was set at $P=0.05$.

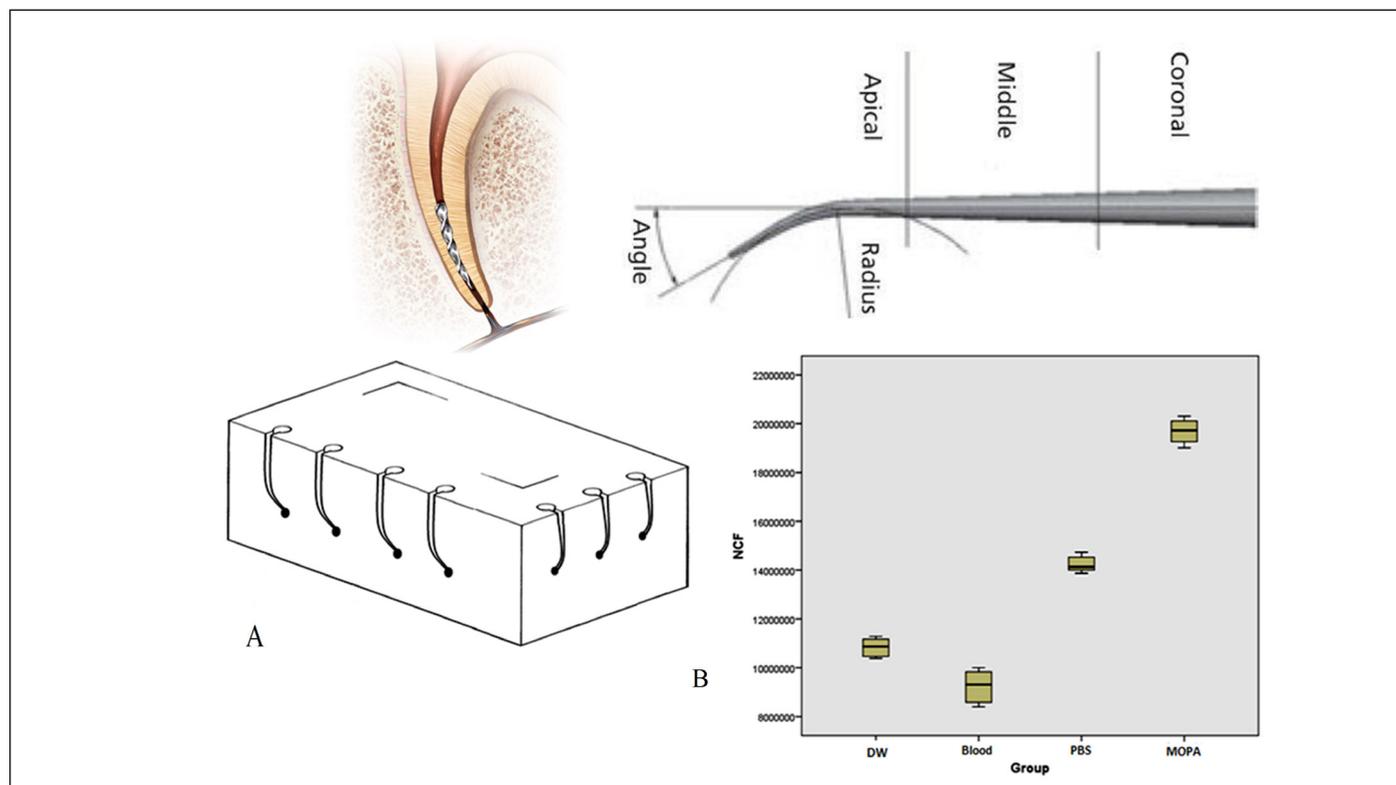


Figure 1. The schematic figure of the stainless-steel block used for NCF evaluation of experimental groups (A). The box plots of the NCF of experimental groups revealing the lowest values in blood group and the highest values in MOPA group (B).

RESULTS

Mean±standard deviations of NCF for experimental groups were 1121884±289811 (DW), 845221±324871 (blood), 1432561±289851 (PBS) and 1978214±662101 (MOPA). Samples in blood and MOPA groups showed significantly the lowest and highest NCF values, respectively ($p<0.05$). Samples in PBS samples showed significantly higher values than DW and blood samples ($p<0.05$) (Figure 1B).

In the DW group, SEM results revealed impurities on the surface with irregular honeycomb pattern. In the blood group, images showed intergranular pattern with aggregation of chloride and nitrogen derived from blood on the fracture surface. In the PBS group, macro dimple pattern was observed, and in the MOPA group, a honeycomb pattern was observed with lower impurities (Figure 2).

DISCUSSION

The separation of endodontic NiTi rotary instruments due to the cyclic or torsional fatigue is an obstacle in endodontic treatments. Although this does not occur all the time, but its occurrence leads to the failure of root canal therapies (26). Among all the reasons for cyclic fatigue failure, the radius of canal curvature is known to have the greatest role in this failure (27). Plotino et al. in their review of the literature on cyclic fatigue testing of NiTi endodontic rotary instruments reported that there are not any approved testing protocols introduced by the International Standard Organisation for this fatigue failure. They also concluded that nearly all studies of

cyclic fatigue were performed in a glass or metal tube (28). In the present study, a stainless-steel block was selected, which after preparation of artificial canals, was subjected to experimental conditions.

The effect of different substances has been discussed on the behaviour of NiTi rotary devices (29). Alapati et al. (29) indicated that scanning electron microscopy observations of clinically failed instruments, suggestions are offered for improving their fracture resistance and can be used as method for fractography analysis. Previous authors have acclaimed that chemical material, such as irrigating solutions, disinfectants and sterilisation process, might have an adverse effect on the structure of NiTi instruments (12, 13). Peters et al. indicated that immersion in NaOCl at temperatures of 21°C and 60°C for 1 or 2 h can reduce cyclic fatigue resistance (30). Cheung et al. (31) investigated effect of different environments on low-cycle fatigue (LCF) of NiTi instruments. These authors addressed deionised water and sodium hypochlorite as corrosive substances, which can drastically influence the LCF behaviour of these instruments (31). The results of present study also showed that deionised water can reduced the cyclic fatigue compared with PBS.

According to SEM images, chloride ion aggregation on the surface of samples stored in blood appears to be an important factor in decreasing of NCF. In a previous study, authors reported that NiTi-made alloys are primarily susceptible to corrosion when they come into contact with solutions that

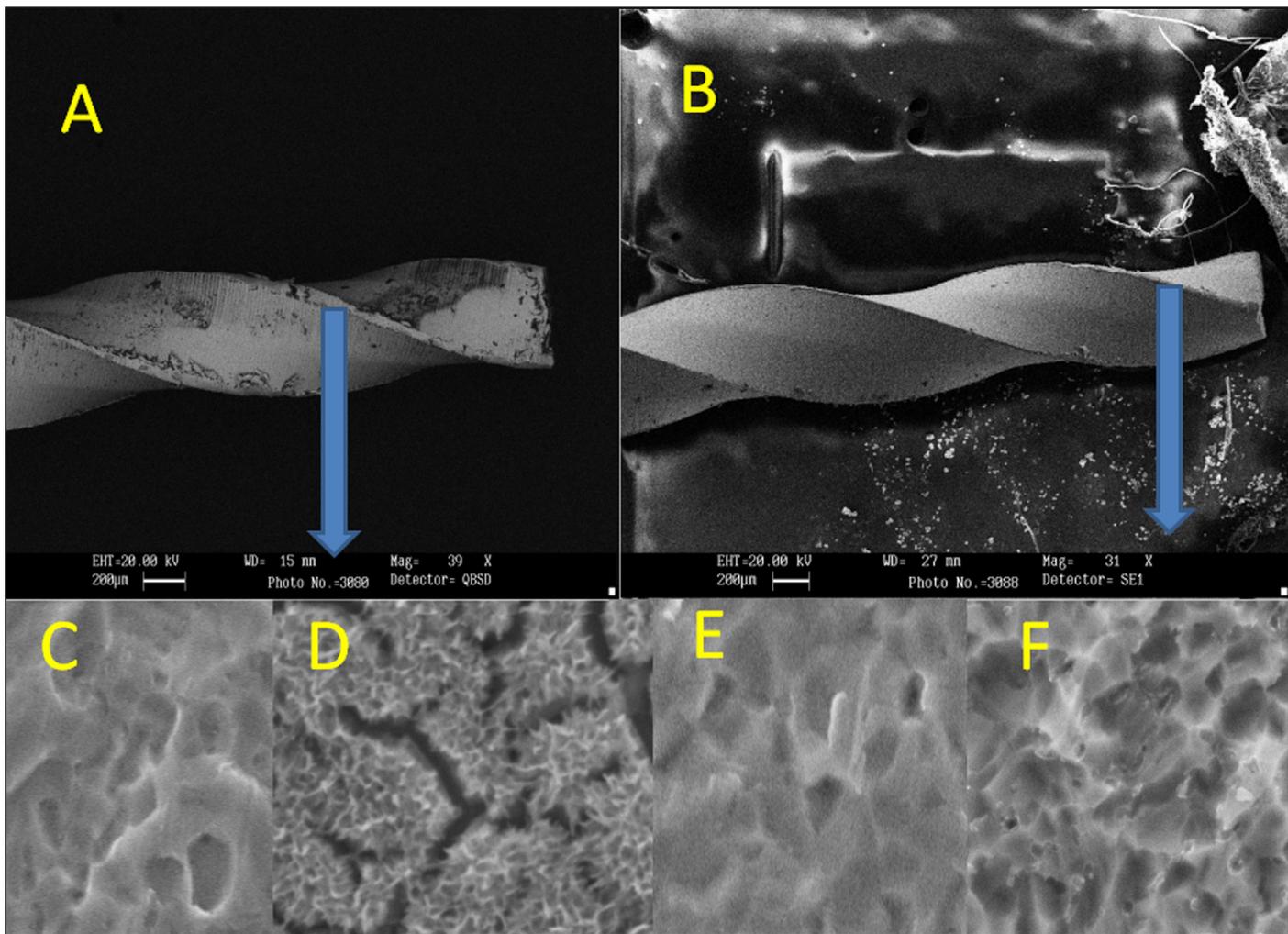


Figure 2. SEM micrographs for samples immersed in blood (39× mag) (A); SEM images of samples immersed in MOPA solution (31× mag) (B); SEM images of deionised water group showing irregular honeycomb pattern (2000× mag) (C); SEM images of blood group with intergranular pattern (2000× mag) (D); SEM images of PBS samples with macro dimple pattern (2000× mag) (E); SEM images of MOPA group specimens revealed honeycomb pattern with lesser impurities on the surface (2000× mag) (F). Please note that the arrows in A and B show the cutting edge of NiTi instruments that were used for further evaluation by 2000× magnification.

contain chlorides (32). Many authors have investigated the effect of chloride ion concentration on crevice corrosion of titanium (33). According to the fact that pitting or crevice corrosion can be regarded as an initiating factor for fatigue failure, it can be presumed that the conventional fatigue failure might be related to the corrosion fatigue. This issue can finally affect the instrument fatigue resistance (12, 13).

Fretting corrosion has been determined as a type of corrosion occurring as a result of oscillatory movements (14). During fretting corrosion, some fluctuations can be observed on the surface of titanium, which includes the removal of the passivating oxide layer (known as depassivation) and production of oxide layer (known as repassivation) at the corroded site that are in a dynamic equilibrium (34-36). One of the most basic limitations of the present study is its exclusive alteration of temperature of rotary systems during the application; therefore, we will aim to check this factor and the effect of failure in future studies. Results of NCF data in MOPA group showed an

increase in cyclic fatigue resistance. This issue can be explained by the anti-corrosive behaviour of MOPA due to the effect of this substance on the oxide layer present on the surface of instrument. It appears that MOPA, by protecting the oxide layer from depassivation, can reduce the fluctuations mentioned above. This phenomenon can result in the reduction of fretting corrosion and finally affect the NiTi file corrosion fatigue resistance. The SEM images of this group also showed lesser impurities, which can be attributed to the protection of oxide layer from fretting and crevice corrosion.

CONCLUSION

1. Deionised water and chloride ions can be regarded as corrosive substances, which affect negatively on the lifetime of NiTi rotary instruments. This is mainly due to the crevice corrosion starting on the surface of instrument followed by fatigue failure and instrument separation. Thus, the use of NiTi rotary files in the bleeding root canals might be threatened in the presence of blood. However, the con-

tact time in root canals is much lower than the storage time considered in this study.

- The tested novel anti-corrosive solution (MOPA) could be suggested as an effective treatment with corrosion-inhibitory action. This substance can increase fatigue resistance and NCF of NiTi rotary files due to stabilisation of the passivating oxide layer. This can reduce the fretting corrosion process, which is considered as an acceleration for crevice corrosion.

Ethics Committee Approval: All the experiments were conducted in accordance with the Association for Research in Vision and Ophthalmology statement for the use of Animals in Ophthalmic and Vision Research and were approved by the Institutional Animal Care and use Committee of the University of Wisconsin School of Medicine and Public health.

Informed Consent: N/A.

Peer-review: Externally peer-reviewed.

Acknowledgements: The authors express special thanks to Regenerative Group of Dr. H Afsar Lajevardi Research Cluster. The work in NS lab is supported by an unrestricted award from Research to Prevent Blindness to the Department of Ophthalmology, Retina Research Foundation, P30 EY016665, P30 CA014520, EPA 83573701, EY026078 and EY022883. NS is a recipient of RPB Stein Innovation Award. This publication is dedicated to the memory of Dr. H. Afsar Lajevardi [37], a legendry Pediatrician (1953-2015) who passed away during this project. We will never forget Dr. H Afsar Lajevardi's kindness and support.

Author Contributions: Concept - M.A.S.; Design- A.A.; Supervision - F.G.G., N.S.; Resources - A.A.; Materials - M.A.S.; Data Collection and/or Processing - M.A.S.; Analysis and/or Interpretation - A.A.; Literature Search - A.A.; Writing Manuscript - M.A.S., A.A.; Critical Review - F.G.G., N.S.

Conflict of Interest: No conflict of interest was declared by the authors.

Financial Disclosure: The authors declared that this study has received no financial support.

REFERENCES

- Civjan S, Huget EF, DeSimon LB. Potential applications of certain nickel-titanium (nitinol) alloys. *J. Dent. Res.* 1975; 54(1): 89-96. [\[CrossRef\]](#)
- Walia HM, Brantley WA, Gerstein H. An initial investigation of the bending and torsional properties of Nitinol root canal files. *J Endod* 1988; 14(7): 346-51. [\[CrossRef\]](#)
- Schafer E, Oitzinger M. Cutting efficiency of five different types of rotary nickel-titanium instruments. *J Endod* 2008; 34(2): 198-200. [\[CrossRef\]](#)
- Yared G. In vitro study of the torsional properties of new and used Profile nickel titanium rotary files. *J Endod* 2004; 30(6): 410-2. [\[CrossRef\]](#)
- Pruett JP, Clement DJ, Carnes DL, Jr. Cyclic fatigue testing of nickel-titanium endodontic instruments. *J Endod* 1997; 23(2): 77-85. [\[CrossRef\]](#)
- Gambarini G. Cyclic fatigue of nickel-titanium rotary instruments after clinical use with low- and high-torque endodontic motors. *J Endod* 2001; 27(12): 772-4. [\[CrossRef\]](#)
- Sattapan B, Nervo GJ, Palamara JE, Messer HH. Defects in rotary nickel-titanium files after clinical use. *J Endod* 2000; 26(3): 161-5. [\[CrossRef\]](#)
- Svec TA, Powers JM. Effects of simulated clinical conditions on nickel-titanium rotary files. *J Endod* 1999; 25(11): 759-60. [\[CrossRef\]](#)
- Bahia MG, Martins RC, Gonzalez BM, Buono VT. Physical and mechanical characterization and the influence of cyclic loading on the behaviour of nickel-titanium wires employed in the manufacture of rotary endodontic instruments. *Int Endontic J* 2005; 38(11): 795-801. [\[CrossRef\]](#)
- Gambarini G, Grande NM, Plotino G, et al. Fatigue resistance of engine-driven rotary nickel-titanium instruments produced by new manufacturing methods. *J Endod* 2008; 34(8): 1003-5. [\[CrossRef\]](#)
- Zinelis S, Darabara M, Takase T, et al. The effect of thermal treatment on the resistance of nickel-titanium rotary files in cyclic fatigue. *Oral surgery, oral medicine, oral pathology, oral radiology, and endodontics* 2007; 103(6): 843-7. [\[CrossRef\]](#)
- Sprowls D. Evaluation of corrosion fatigue. ASM International, ASM Handbook. 1987; 13: 291-302.
- Darabara M, Bourithis L, Zinelis S, Papadimitriou GD. Susceptibility to localized corrosion of stainless steel and NiTi endodontic instruments in irrigating solutions. *Int Endontic J* 2004; 37(10): 705-10. [\[CrossRef\]](#)
- Thull R, Schaldach M. Corrosion of highly stressed orthopedic joint replacements. In: *Engineering in Medicine*. Springer; 1976. p. 242-56. [\[CrossRef\]](#)
- Meunier A, Sedel L. Actes des journees francophones de tribologie-corrosion (JFTC 1998). Société Tribologique de France, SIRPE, Paris, France 1998: 193-200.
- Brown SA, Flemming CA, Kawalec JS, et al. Fretting corrosion accelerates crevice corrosion of modular hip tapers. *Journal of applied biomaterials: an official journal of the Society for Biomaterials* 1995; 6(1): 19-26. [\[CrossRef\]](#)
- Dhouibi L, Triki E, Raharinaivo A. The application of electrochemical impedance spectroscopy to determine the long-term effectiveness of corrosion inhibitors for steel in concrete. *Cement and Concrete Composites* 2002; 24(1): 35-43. [\[CrossRef\]](#)
- Liang H, Li L, Poor N, Sagüés A. Nitrite diffusivity in calcium nitrite-admixed hardened concrete. *Cement and Concrete Research* 2003; 33(1): 139-46. [\[CrossRef\]](#)
- de Rincon OT, Pérez O, Paredes E, et al. Long-term performance of ZnO as a rebar corrosion inhibitor. *Cement and concrete composites* 2002; 24(1): 79-87. [\[CrossRef\]](#)
- Cigna R, Familiari G, Gianetti F, Proverbio E. Corrosion and Corrosion Protection of Steel in Concrete. Sheffield, UK 1994; 848.
- Bjegovic D, Sipos L, Ukrainczyk V, Miksic B. Diffusion of the MCI 2020 and 2000 corrosion inhibitors into concrete, *Corrosion and Corrosion Protection of Steel in Concrete*. Ed. RN Swamy, Sheffield Academic Press, Sheffield 1994.
- Elsener B. In: *Proceedings of the COST 521 Workshop v, Belfast, 2000*.
- Saghiri MA, Sheibani N, Asatourian A, Garcia-godoy F. ANTICORROSIVE AND ANTI-FATIGUE CHEMICAL COMPOSITION FOR NICKEL-TITANIUM DENTAL INSTRUMENTS AND A METHOD OF SYNTHESIZING THE SAME. In: *US Patent 20,160,024,311*; 2016.
- Huntsman Corporation Technical bulletin. No. 5011-0408 MM -, 3 pages. Available from: www.huntsman.com/MOPA.pdf
- Grande N, Plotino G, Falanga A, Somma F. A new device for cyclic fatigue testing of Niti rotary endodontic instruments. *Int Endontic J* 2005;38(12):938.
- Spili P, Parashos P, Messer HH. The impact of instrument fracture on outcome of endodontic treatment. *J Endod* 2005;31(12):845-50. [\[CrossRef\]](#)
- Haikel Y, Serfaty R, Bateman G, et al. Dynamic and cyclic fatigue of engine-driven rotary nickel-titanium endodontic instruments. *J. Endod.* 1999;25(6):434-40. [\[CrossRef\]](#)
- Plotino G, Grande NM, Cordaro M, et al. A review of cyclic fatigue testing of nickel-titanium rotary instruments. *J Endod* 2009;35(11):1469-76. [\[CrossRef\]](#)
- Alapati SB, Brantley WA, Svec TA, et al. SEM observations of nickel-titanium rotary endodontic instruments that fractured during clinical use. *J. Endod.* 2005;31(1):40-3. [\[CrossRef\]](#)

30. Peters OA, Roehlike JO, Baumann MA. Effect of immersion in sodium hypochlorite on torque and fatigue resistance of nickel-titanium instruments. *J Endod* 2007;33(5):589-93. [\[CrossRef\]](#)
31. Cheung GS, Shen Y, Darvell BW. Effect of environment on low-cycle fatigue of a nickel-titanium instrument. *J Endod* 2007;33(12):1433-7. [\[CrossRef\]](#)
32. Li X, Wang J, Han E-h, Ke W. Influence of fluoride and chloride on corrosion behavior of NiTi orthodontic wires. *Acta Biomaterialia* 2007;3(5):807-15. [\[CrossRef\]](#)
33. Satoh H, Shimogori K, Kamikubo F. The crevice corrosion resistance of some titanium materials. *Platinum Metals Review* 1987;31(3):115-21.
34. Berradja A, Bratu F, Benea L, et al. Effect of sliding wear on tribo-corrosion behaviour of stainless steels in a Ringer's solution. *Wear* 2006;261(9):987-93. [\[CrossRef\]](#)
35. Tang B, Wu PQ, Fan AL, et al. Improvement of Corrosion-Wear Resistance of Ti-6Al-4V Alloy by Plasma Mo-N Surface Modification. *Advanced engineering materials* 2005;7(4):232-8. [\[CrossRef\]](#)
36. Quan Z, Wu P-Q, Tang L, Celis J-P. Corrosion-wear monitoring of TiN coated AISI 316 stainless steel by electrochemical noise measurements. *Appl. Sci. Res.* 2006; 253(3): 1194-7. [\[CrossRef\]](#)
37. M.A. Saghiri, A.M. Saghiri In Memoriam: Dr. Hajar Afsar Lajevardi MD, MSc, MS (1955–2015) *Iran J. Pediatr.*, 27 (2017), p. e8093 <http://dx.doi.org/10.5812/ijp.8093> [\[CrossRef\]](#)