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Original Article

Effect of sodium hypochlorite on conventional and heat-treated nickel-titanium endodontic rotary instruments — An *in vitro* study

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Abstract *Background/purpose:* Sodium hypochlorite (NaOCl) is a highly alkaline solution which exhibits antimicrobial properties. However, it causes corrosion to endodontic rotary files. This study investigated the effect of NaOCl on the metal surface of five different unused endodontic rotary files *in vitro*.
Materials and methods: Two non-heat-treated nickel-titanium (NiTi) files, ProTaper, BioRace, and three heat-treated NiTi files, VortexBlue, TRUShape, and EdgeFile X7 files, were immersed in 4% NaOCl for 5, 10, and 20 min, and 1, 6 and 24 h. The corrosion susceptibility was evaluated by visual inspection and scanning electron microscopy (SEM).
Results: In the TRUShape group, the black particulate matter was mostly formed at the file's curvature and shaft. A large amount of precipitate accumulated in the EdgeFile group. The extent and prevalence of surface defects were found to be consistently higher in EdgeFile X7 instruments than in any other instruments. EdgeFile X7 and TRUShape files exhibited a greater corrosive tendency to NaOCl than BioRace, ProTaper, and VortexBlue not only under visual inspection, but also under SEM analysis after prolonged immersion (1, 6, 24 h) in 4% NaOCl. However, shorter immersion periods (5, 10, 20 min) showed little surface corrosion across all experimental groups.
Conclusion: Within the limitations of this study, EdgeFile X7 and TRUShape files exhibit greater corrosive tendencies to NaOCl *in vitro* than BioRace, ProTaper, and VortexBlue files after prolonged immersion in 4% NaOCl. However, shorter immersion periods, which more closely approximate clinical conditions during single root canal therapy, may not show surface corrosion across all experimental groups.

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Introduction

The primary goal of root canal treatment is to optimize disinfection of the root canal system to prevent or eliminate apical periodontitis.[1] Root canal disinfection is achieved through chemo-mechanical instrumentation, which includes mechanical debridement of the root canal walls by using endodontic files and chemical irrigation with sodium hypochlorite (NaOCl) to dissolve organic substances and eliminate microbes in the root canal space.[2,3] The metallurgy of endodontic files has evolved over the decades. Endodontic files have changed from traditional stainless steel to nickel-titanium (NiTi) alloys,[4] which exhibit greater physical properties, including flexibility, high torsional strength, and shape memory capabilities.[4,5] Furthermore, since early 2000, heat-treated NiTi endodontic files produced with proprietary methods consisting of drawing the raw wire under specific tension and heat treatments at various temperatures have been introduced. The heat treatment of NiTi instruments can induce changes in the transformation behavior of the metal alloy, which provides higher fatigue resistance, flexibility, and cutting efficiency compared with traditional superelastic NiTi and stainless steel files.[6–8] These enhanced physical properties of the heat-treated endodontic files may enable more efficient root canal preparation by facilitating instrumentation in curved canals and maintaining the file in the center of root canal space.[9,10]

Due to the anatomical complexity of the root canal system, mechanical debridement alone has limitations in removing infected tissues and microbes present in isthmuses and ramifications of the root canal system. Therefore, the use of irrigation solutions in conjunction with mechanical instrumentation is essential for successful root canal treatment.[11] The most widely used irrigant is NaOCl, commonly prepared at various concentrations (0.5%–5.5%). It has a unique tissue-dissolving property,[11,12] as well as a broad antimicrobial spectrum that is both bactericidal and virucidal.[13–15]

During chemo-mechanical instrumentation of the root canal system, endodontic files are constantly in contact with NaOCl inside the root canal space. Furthermore, throughout the lifetime of an endodontic file, it may need to undergo multiple cycles of cleaning and disinfection after each use. NaOCl is a highly alkaline solution and has been shown to cause measurable corrosion at concentrations of 5–5.25%[16] to both stainless steel and NiTi files.[16,17] The effect of corrosion on files has been discussed in the literature. As NaOCl selectively removes nickel from the instrument's surface, immersion in NaOCl leads to micro-pitting, which negatively affects the physical and mechanical properties of NiTi files.[18] Various studies have investigated the corrosive effects of NaOCl on both conventional NiTi and stainless steel endodontic instruments,[19] using microscopy, electrochemical analysis, and by measuring Ni and Ti release.[16,20,21] However, so far few studies have assessed the effects of NaOCl on the surface of heat-treated NiTi instruments.

The aim of this study was to evaluate and compare *in vitro* the corrosion susceptibility of five Ni–Ti endodontic instruments immersed in 4% NaOCl both by visual inspection and by scanning electron microscopy (SEM).

Materials and methods

Selection and preparation of the samples

Five endodontic file systems were used for this study: ProTaper (Dentsply Tulsa Dental Specialties, Tulsa, OK, USA), BioRace (FKG Dentaire, La Chaux-de-Fonds, Switzerland), VortexBlue (Dentsply Tulsa Dental Specialties), TRUShape (Dentsply Tulsa Dental Specialties), and EdgeFile X7 (EdgeEndo, Albuquerque, NM, USA). A total of 210 new rotary files (42 files from each file system) were used in this study, and each selected file was 25 mm in length, with an ISO #40 tip size. Each file was selected at random, and its rubber stopper was gently removed to avoid causing damage to the file surfaces.

Sodium hypochlorite (NaOCl) treatment

Flat-top 2.0-mL polypropylene microtubes (Fisher Scientific, Hampton, NH, USA) were filled with 1.0 mL of 4% NaOCl. Each file was placed in an individual tube and approximately 18 mm of the file, including the flute and shaft, was immersed in the NaOCl solution. The lid of the tube was left open. The handle of the file was left unexposed. All microtubes were placed in an incubator at 37 °C during the experimental times to simulate the temperature at which NaOCl is found in the root canals in a clinical situation. To detect the visual changes during the 24 h-observation, all samples were visualized with the naked eye against a white background. The microtubes were monitored every 5 min for one hour and then every hour for 5 h and at the 24th hour, and the samples were evaluated for color changes in the solutions, the presence of bubbles, and precipitate formation. A total of 18 files from each file system were subjected to NaOCl treatment. File immersion in NaOCl was terminated at six time points, namely 5, 10, and 20 min, and 1, 6 and 24 h. After examining the files at each time point, three files were taken out of each microtube and prepared for further analysis. This time-course study was independently repeated two times, resulting in a total of 36 files to be examined for each file system. Furthermore, additional six files from each file system were chosen at random and were left unexposed to NaOCl to serve as negative controls.

Evaluation by scanning electron microscopy (SEM)

After being immersed in 4% NaOCl for each experimental period, each file was taken out of the microtube, and rinsed in distilled water twice, and allowed to air-dry at room temperature overnight. Three files from each file system were randomly selected among the six files from each time point of immersion in NaOCl. Selected files were then mounted on an SEM stub and the surfaces were examined using SEM. Furthermore, six unused rotary files (negative controls) from each file system were screened for any defects using SEM. The Quanta 600 Environmental SEM (FEI Co., Hillsboro, OR, USA) operated at 15 kV acceleration voltage was used for analysis of the samples. SEM images of the shaft, middle, and tip (end) portions of each file were taken at $\times 100 - \times 150$ magnifications and analyzed.

Statistical analysis

Multiple comparisons were performed using the chi-square test. Differences at $P < .05$ were considered significant.

Results

Visual inspection of the file samples in sodium hypochlorite

The following general features were observed during the corrosion process. Bubbles were formed on the metal surface of the immersed file. The amount of bubble formation increased over time and was found to be most abundant in the BioRace, TRUShape, and EdgeFile groups. Furthermore, the volume of sodium hypochlorite decreased over time as the corrosion process proceeded. The remaining sodium hypochlorite solution changed its color from colorless with a yellowish tint to a grayish, purplish color in the most corroded groups, namely TRUShape and EdgeFile.

The formation of black particulate both on the file surface and as a precipitation product was the most significant observation during the corrosion process. No corrosion products were observed in any of the five groups after a 15 min-immersion in NaOCl. At 20 min, all files in the EdgeFile group showed black particulate matter, while in the TRUShape group, the first sign of corrosion was detected at one hour. The amount of black particulate formation increased over time for both EdgeFile and TRUShape (Fig. 1). Although there were variations in black particulate formation within each brand of files, the following general patterns were observed. In the TRUShape group, the black particulate matter was mostly formed at the bulb of the file, i.e. at the file's curvature, and at the shaft. In the EdgeFile group, on the other hand, most of the black precipitate was formed on the shaft of the file with the accumulation of large amount of precipitate (Fig. 1A). At the end of the observation period (24 h), the black particulate matter was also observed on the shaft of some of the BioRace files, but not all (Fig. 1A). The EdgeFile groups showed a significantly higher tendency of corrosion than other file groups at every time point ($P < .001$, Fig. 1B). After 24 h-immersion in NaOCl, the percentages of files with precipitate formation were ProTaper (0%), VortexBlue (0%), BioRace (33.3%), TruShape (83.3%), and EdgeFile (100%).

SEM analysis

The SEM results of the 20-min-, 6-h-, and 24-h immersion groups were included as these time points represented the most significant visual changes as a result of the corrosive effect of NaOCl. The most representative SEM images of the general features at these time points were presented in Fig. 2.

1) Control group (non-exposed files)

Screening of the six unused, brand-new files from each group under SEM inspection showed that no single file system was completely free of defects or debris. Pitting was the most common defect on unused files and was evident from the cutting head to the end of the cutting surface (Fig. 2A, white arrows). All EdgeFile X7 files showed black residues on the surface of the working portion of the file (Fig. 2A).

2) 20 min-immersion group

Compared to the control group, no apparent signs of corrosion were identified on the file surface in any of the file groups after 20 min of 4% NaOCl immersion under SEM (Fig. 2B). In the EdgeFile file group, however, a small amount of black precipitate was detectable at the bottom of the microtube at the 20-min direct visual inspection.

3) 6 h-immersion group

While no sign of corrosion was detected in the ProTaper, BioRace, and VortexBlue groups (Fig. 2C), the TRUShape group showed variable evidence of corrosion on the cutting heads and at the S-shaped curves of the files. In all EdgeFile groups, surface pitting, metal flesh, and precipitated corrosion products were detected on the entire surface of the files (Fig. 2C).

4) 24 h-immersion group

ProTaper and VortexBlue files consistently showed no detectable sign of corrosion (Fig. 2D), whereas a small amount of corrosion was detected on the shaft of BioRace files. The TRUShape group showed variable evidence of corrosion on the shaft and flute of the files, and more severe corrosion was identified on the cutting head. An extensive flocculent white precipitate, sometimes overlying a dense black precipitate, was detected on the entire surface of all EdgeFile instruments (Fig. 2D).

Discussion

While several studies have discussed the corrosive effects of NaOCl on conventional NiTi endodontic rotary instruments, few studies have examined the effects of NaOCl on heat-treated NiTi files. In the present study, the effects of NaOCl solution on non-heat-treated, conventional NiTi rotary files (ProTaper, BioRace) and heat-treated NiTi rotary files (VortexBlue, TRUShape, and EdgeFile X7) were investigated by using direct visual inspection and SEM

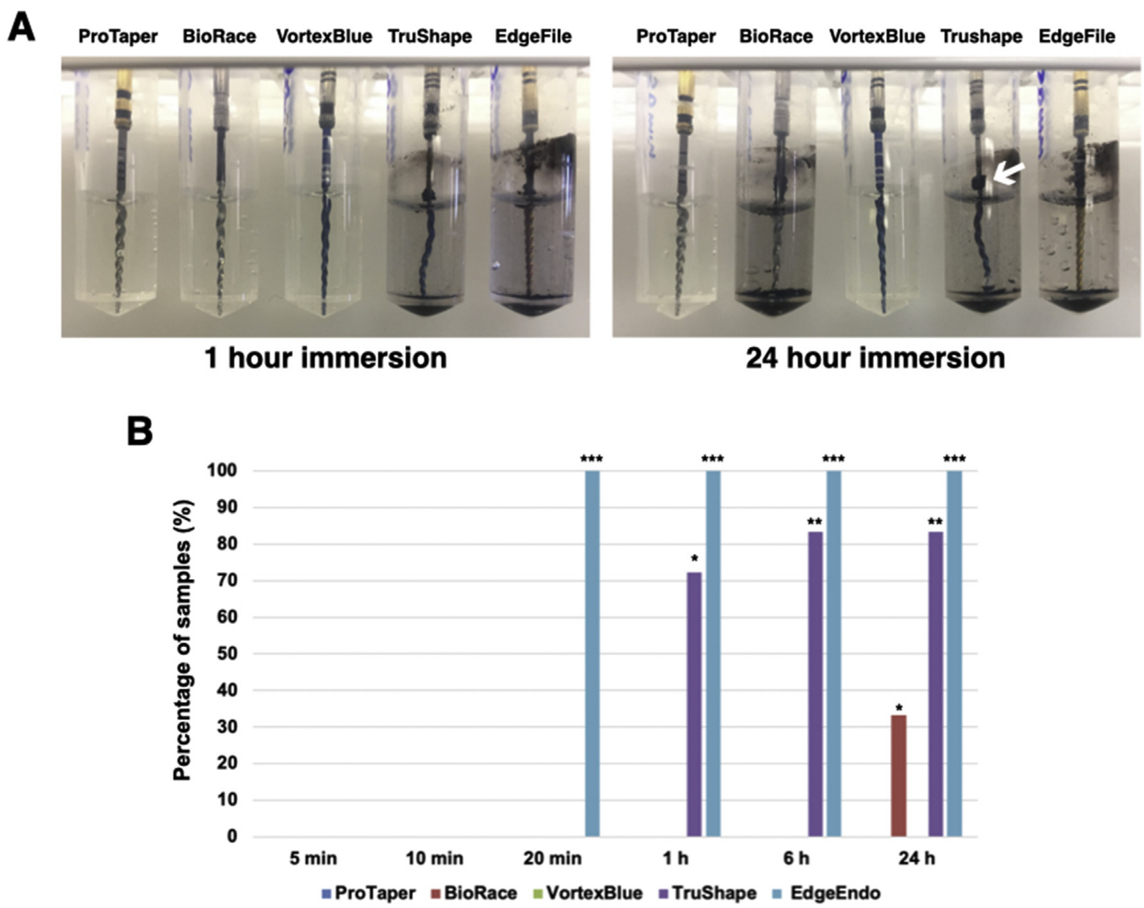

Figure 1 (A) Visual aspect of the interactions between the files and NaOCl. ProTaper, BioRace, VortexBlue, TRUShape, and EdgeFile X7 were immersed in 4% NaOCl for 1 h and 24 h. (B) The percentage of files with precipitate formation after immersing in 4% NaOCl for 5, 10, and 20 min, and 1, 6 and 24 h. *$P < .05$, **$P < .01$, and ***$P < .001$ indicated significance.

analysis of the file surface. Furthermore, this study aimed to determine whether heat-treated NiTi endodontic files are more susceptible to NaOCl corrosion.

Our observation of the black precipitate produced after prolonged periods of NaOCl immersion was consistent with the findings reported by O'Hoy's et al.[20] According to our direct visual analysis of corrosion, the rankings of corrosion resistance of the five endodontic file systems under our testing conditions, from most resistant to least, were as follows: VortexBlue, ProTaper, BioRace, TRUShape, and EdgeEndo X7. The present study found that VortexBlue exhibited greater corrosion resistance to NaOCl than the other two non-heat-treated files. This finding indicates that heat treatment alone does not necessarily increase the file's susceptibility to NaOCl corrosion. Various manufacturing factors, such as file surface treatment after carving (e.g. BioRace's electropolishing), file cross-section geometry, and design (e.g. TRUShape's S-shape design), may all contribute to a discrepancy in the file's susceptibility to NaOCl corrosion. In addition, the visible blue titanium oxide surface layer that is the proprietary manufacturing feature of VortexBlue may be responsible for the file's increased resistance to NaOCl corrosion.

A previously published study reported that VortexBlue exhibits better physical and mechanical properties compared to the other non-heat-treated instrument of the same file design.[10] The specific oxide surface layer manufacturing process may have contributed to the enhanced cyclic fatigue resistance of VortexBlue.[10] With respect to the other mechanical properties of the instrument, namely torsional moment, maximum angular deflection, maximum bending moment, and permanent angular deflection, Haikel et al. evaluated these four mechanical properties as a function of corrosion resistance with NaOCl.[22] They concluded that the mechanical properties of the four conventional NiTi instruments that they examined were not affected by NaOCl, nor was their cutting efficiency.[22] Even though few studies have investigated the various mechanical properties of heat-treated NiTi files after NaOCl immersion, Pedulla et al. have shown that the effect of NaOCl on the cyclic fatigue resistance of heat-treated files was not significant.[18] Future studies should be directed in this area in order to explore the relationships between the effects of NaOCl on the heat-treated file's metal surface and the affected heat-treated file surface's impact on the file's various mechanical properties.

The SEM analysis not only provides a more accurate assessment of the effects of NaOCl immersion, but it also serves as a screening tool for defects on brand-new, unused rotary files. It is important to point out that although none

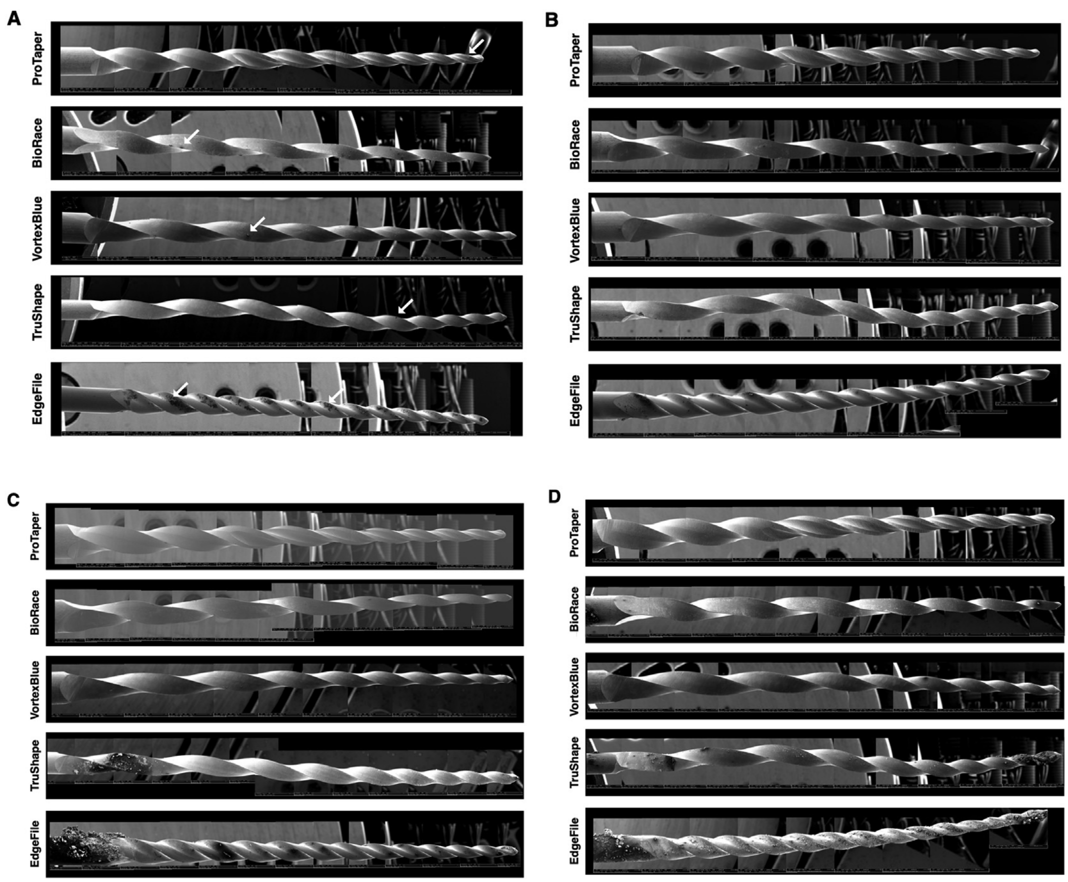

Figure 2 Representative scanning electron microscopic images showing selected samples from shaft, middle, and tip portion of files (magnification ×100 – ×150). (A) Untreated files. The *arrows* indicate pitting. (B) Files after soaking in 4% NaOCl for 20 min. (C) Files after soaking in 4% NaOCl for 6 h. (D) Files after soaking in 4% NaOCl for 24 h.

of the unused files showed their cutting surfaces completely free of defects or debris under SEM examination, the extent and prevalence of surface defects were found to be consistently and significantly higher in EdgeFile X7 (Fig. 2). It is plausible to assume that the lack of adequate quality control and surface treatment standards may lead to the production of instruments with more surface defects and irregularities during manufacturing and induce a corrosion pit that was associated with crack initiation.[23] Nevertheless, the SEM analysis has its limitations, since it can only examine one side of the file at a time. The true extent of corrosion would have been more accurately assessed if both sides of the file were to be examined. This could explain why little evidence of corrosion was identified on the file surface in all experimental groups at 20 min under SEM, even though minute amounts of black particulate were found on the file surface and at the bottom of the microtube in the EdgeFile X7 group by direct visual inspection.

The results of the present study showed that the two heat-treated instruments, TRUShape and EdgeFile X7, are more susceptible to NaOCl. The manufacturers of these three heat-treated file systems recommend single patient use of their rotary files to reduce the risks of cross-contamination and instrument breakage.[24–26] However, it remains unclear whether instrument breakage is a direct result of changes to the metal surface due to NaOCl corrosion in these heat-treated files. Whether our findings may be clinically relevant *in vivo* depends on the number of times of the files' reuse, disinfection control protocol, such as immersing files in 6% NaOCl for 5 min before use,[27] and autoclave sterilization which cumulatively prolongs file contact with NaOCl.[20,28] Therefore, multiple uses of these endodontic files may increase the risk of instrument breakage.

In conclusion, EdgeFile X7 and TRUShape files exhibit a greater corrosive tendency to NaOCl *in vitro* than BioRace, ProTaper, and VortexBlue files after prolonged immersion (1, 6, 24 h) in 4% NaOCl. However, shorter immersion periods (5, 10, 20 min), which more closely approximate clinical conditions during one single root canal therapy, seem to show no surface corrosion across any of the experimental groups. Heat treatment of the NiTi file may not have a direct impact on file's susceptibility to NaOCl corrosion.

Declaration of competing interest

The authors have no conflict of interest relevant to this article.

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References

1. Ørstavik D, Pitt Ford TR, eds. *Essential endodontology. Prevention and treatment of apical periodontitis*. Oxford: Blackwell Science, 1998:1—8.
2. Bystrom A, Sundqvist G. Bacteriologic evaluation of the effect of 0.5 percent sodium hypochlorite in endodontic therapy. *Oral Surg Oral Med Oral Pathol* 1983;55:307—12.
3. Bystrom A, Claesson R, Sundqvist G. The antibacterial effect of camphorated paramonochlorophenol, camphorated phenol and calcium hydroxide in the treatment of infected root canals. *Endod Dent Traumatol* 1985;1:170—5.
4. Glossen CR, Haller RH, Dove SB, del Rio CE. A comparison of root canal preparations using Ni-Ti hand, Ni-Ti engine-driven, and K-Flex endodontic instruments. *J Endod* 1995;21:146—51.
5. Walia HM, Brantley WA, Gerstein H. An initial investigation of the bending and torsional properties of nitinol root canal files. *J Endod* 1988;14:346—51.
6. Shen Y, Zhou HM, Zheng YF, Peng B, Haapasalo M. Current challenges and concepts of the thermomechanical treatment of nickel-titanium instruments. *J Endod* 2013;39:163—72.
7. Gao Y, Gutmann JL, Wilkinson K, Maxwell R, Ammon D. Evaluation of the impact of raw materials on the fatigue and mechanical properties of ProFile Vortex rotary instruments. *J Endod* 2012;38:398—401.
8. Hayashi Y, Yoneyama T, Yahata Y, et al. Phase transformation behaviour and bending properties of hybrid nickel-titanium rotary endodontic instruments. *Int Endod J* 2007;40:247—53.
9. Yahata Y, Yoneyama T, Hayashi Y, et al. Effect of heat treatment on transformation temperatures and bending properties of nickel-titanium endodontic instruments. *Int Endod J* 2009;42:621—6.
10. Plotino G, Grande NM, Cotti E, Testarelli L, Gambarini G. Blue treatment enhances cyclic fatigue resistance of vortex nickel-titanium rotary files. *J Endod* 2014;40:1451—3.
11. Zehnder M. Root canal irrigants. *J Endod* 2006;32:389—98.
12. Hand RE, Smith ML, Harrison JW. Analysis of the effect of dilution on the necrotic tissue dissolution property of sodium hypochlorite. *J Endod* 1978;4:60—4.
13. Best M, Springthorpe VS, Sattar SA. Feasibility of a combined carrier test for disinfectants: studies with a mixture of five types of microorganisms. *Am J Infect Control* 1994;22:152—62.
14. Rutala WA. APIC guideline for selection and use of disinfectants. 1994, 1995, and 1996 APIC Guidelines Committee. Association for Professionals in Infection Control and Epidemiology, Inc. *Am J Infect Control* 1996;24:313—42.
15. Pashley EL, Birdsong NL, Bowman K, Pashley DH. Cytotoxic effects of NaOCl on vital tissue. *J Endod* 1985;11:525—8.
16. Stokes OW, Fiore PM, Barss JT, Koerber A, Gilbert JL, Lautenschlager EP. Corrosion in stainless-steel and nickel-titanium files. *J Endod* 1999;25:17—20.
17. Edie JW, Andreasen GF, Zaytoun MP. Surface corrosion of nitinol and stainless steel under clinical conditions. *Angle Orthod* 1981;51:319—24.
18. Pedulla E, Benites A, La Rosa GM, et al. Cyclic fatigue resistance of heat-treated nickel-titanium instruments after immersion in sodium hypochlorite and/or sterilization. *J Endod* 2018;44:648—53.
19. Peters OA, Roehlike JO, Baumann MA. Effect of immersion in sodium hypochlorite on torque and fatigue resistance of nickel-titanium instruments. *J Endod* 2007;33:589—93.
20. O'Hoy PY, Messer HH, Palamara JE. The effect of cleaning procedures on fracture properties and corrosion of NiTi files. *Int Endod J* 2003;36:724—32.
21. Busslinger A, Sener B, Barbakow F. Effects of sodium hypochlorite on nickel-titanium Lightspeed instruments. *Int Endod J* 1998;31:290—4.
22. Haikel Y, Serfaty R, Wilson P, Speisser JM, Allemann C. Mechanical properties of nickel-titanium endodontic instruments and the effect of sodium hypochlorite treatment. *J Endod* 1998;24:731—5.
23. Cheung GSP, Shen Y, Darvell BW. Does electropolishing improve the low-cycle fatigue behavior of a nickel-titanium rotary instrument in hypochlorite? *J Endod* 2007;33:1217—21.
24. Vortex Blue File. *Directions for use*. Available at: https://www.dentsplysirona.com/content/dam/dentsply/pim/manufacturer/Endodontics/Glide_Path__Shaping/Rotary__Reciprocating_Files/Shaping/Vortex_Blue_Rotary_Files/Vortex-Blue-ayg107i-en-1402. [Accessed 2 October 2017].
25. TRUShape File. *Directions for use*. Available at: https://www.dentsplysirona.com/content/dam/dentsply/pim/manufacturer/Endodontics/Glide_Path__Shaping/Rotary__Reciprocating_Files/3D_Conforming/TRUShape_3D_Conforming_Files/TRUShape-3D-Files-Rev-2-DFU-EN-camrdnr-en-1506.pdf. [Accessed 2 October 2017].
26. EdgeFile X-7 Files. *Directions for use*. Available at: https://edgeendo.com/wp-content/uploads/2015/08/DFU-EdgeFile-x7.pdf. [Accessed 2 October 2017].
27. Roth TP, Whitney SI, Walker SG, Friedman S. Microbial contamination of endodontic files received from the manufacturer. *J Endod* 2006;32:649—51.
28. Nair AS, Tilakchand M, Naik BD. The effect of multiple autoclave cycles on the surface of rotary nickel-titanium endodontic files: An in vitro atomic force microscopy investigation. *J Conserv Dent* 2015;18:218—22.