

Effect of Immersion in Sodium Hypochlorite on Torque and Fatigue Resistance of Nickel-Titanium Instruments

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Abstract

This study investigated the effect of immersion in sodium hypochlorite (NaOCl) on torque and fatigue resistance of two nickel-titanium files. Size 25 .04 ProFile and RaCe files were immersed in 5.25% NaOCl for 1 or 2 hours at temperatures of 21°C and 60°C. Torque and angle at failure were measured according to ISO 3630-1. Resistance to cyclic fatigue was determined by counting rotations to breakage in a 90° curve with a 5-mm radius. Data were analyzed by 2-way analysis of variance. Torsional resistance of both rotaries was not significantly affected by immersion in NaOCl, except after 2 hours of immersion at 60°C. Resistance to cyclic fatigue decreased significantly for ProFile and RaCe instruments after immersion in NaOCl. Spontaneous fractures occurred in 28 of 160 files during immersion in NaOCl. In conclusion, nickel-titanium rotaries have reduced resistance to cyclic fatigue after contact with heated NaOCl and may then be considered single-use instruments. (*J Endod* 2007;33:589–593)

Key Words

Corrosion, fatigue, nickel-titanium, sodium hypochlorite, torque

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The use of sodium hypochlorite (NaOCl) to irrigate root canals is currently the gold standard to achieve tissue dissolution and disinfection (1, 2). It has been shown that the amount of available chlorine is important for the tissue dissolving and antibacterial effect of NaOCl (3, 4). Several mechanisms have been suggested to increase that amount (5): the use of undiluted NaOCl (5.25% v/v) (6), increasing volume turnover (7), or heating NaOCl solutions (8). Immersion in 1% NaOCl at 60°C results in significantly more effective tissue dissolution compared with a 5.25% solution. Similarly, an increased killing capacity of *Enterococcus faecalis* was observed (8).

Before irrigating the root canal system, canals are enlarged and appropriately shaped; over the last two decades, nickel-titanium (NiTi) rotary files have become increasingly popular for this task (9). There is clear experimental and clinical evidence that the use of NiTi files, compared with stainless-steel K-files, promotes canal shapes that follow the original canal path with less procedural errors (9, 10). On the other hand, in vitro studies have suggested that NiTi rotary files fracture more often than stainless-steel files (11).

Two mechanisms that may lead to NiTi rotary fracture, namely cyclic fatigue and torsional overloading, have been described (12). Cyclic fatigue is a result of rotation around a curve with the consequence of repeated extension and compression of metal and finally work hardening followed by fracture (13). Torsional fracture may occur from overzealous apically directed pressure and wedging the files' tip into the canal, resulting in excess friction and then fracture (12).

Efforts are ongoing to identify factors that impact NiTi rotary fracture resistance (e.g., file sequence, torque-limiting motors, cross-sectional file design, and file surface finish). Electropolishing and ion deposition are two techniques to modify NiTi surfaces that may have an effect on physical properties (14). One additional factor potentially limiting the resistance to fatigue and torsional fracture is corrosion that may occur in the presence of NaOCl solution (15–17).

Sodium hypochlorite is present in root canals before inserting any rotary file to provide disinfection but also lubrication (18). The time course and extent of action of NaOCl on NiTi surfaces is currently unclear. This partly stems from the fact that NaOCl is used for canal disinfection but also for cleaning of used rotary files to remove organic matter (19). Significant signs of surface corrosion were reported by O'Hoy and colleagues (15) after overnight immersion, whereas Darabara et al. (16) found no signs of corrosion after immersion for 1 hour in 5.25% NaOCl and 17% ethylenediamine-tetraacetic acid (EDTA) heated to 37°C.

If present, corrosion may negatively affect physical properties of NiTi files (15, 20). Therefore, the aim of the current study was to assess resistance to fatigue and torque of two brands of NiTi files after immersion in heated NaOCl solutions. The impact of electropolishing on corrosion resistance was also investigated.

Materials and Methods

A total of 200 unused ProFile (Dentsply Maillefer, Ballaigues, Switzerland) and RaCe (FKG, La Chaux-de-Fonds, Switzerland) NiTi files size 25 .04 taper were used in this study. Files were randomly assigned to 1 control group and 4 experimental groups ($n = 20$ each); files in the latter were then immersed in 5.25% NaOCl (Dan Klorix; Colgate Palmolive, Hamburg, Germany). For that purpose, files were placed into individual glass vials for 1 or 2 hours. Vials were either stored at room temperature ($21^\circ \pm 2^\circ\text{C}$) or were placed in an electronically controlled heating device (Multiblock 2001;

Labline, Mehare Park, IL) after the preset immersion temperature of $60^{\circ} \pm 1^{\circ}\text{C}$ had been reached. All files were wiped dry immediately after removal from immersion with cotton swabs, air dried, and tested.

Torque and Fatigue Testing

Torsional fracture loads were determined for control and experimental rotary files according to International Organization for Standardization (ISO) 3630-1 (21) by using a testing device described in detail elsewhere (12, 22). Briefly, 3 mm of file tips were secured firmly in a specifically designed soft brass holder. The files' shank was then rotated at 2 rpm until fracture occurred. Care was taken to adjust files into the holder so that no tension or deflection was present before testing.

Torque and angle of rotation were continuously measured with a memocouple (MTTRA 2 with amplifier Microtest; Microtec Systems, Villingen, Germany) and digitized at 24 Hz (PCI-MIO-16XE; National Instruments, Austin, TX). Data were then fed into a computer (G3 Power Mac; Apple, Cupertino, CA) running *Endotest*, a custom-made software package. *Endotest* controls rotational speed and axial movement of the tested rotary instrument and records the parameters insertion depth, torque, force, and number of rotations in real time.

Torque at failure (in Nmm) and angle at failure (in degrees) were calculated offline from original records and tabulated. The testing device was frequently calibrated by using custom-made brass weights and levers; torque measurement accuracy was 0.5%.

To determine resistance to fatigue, numbers of rotations to fracture were counted by using a tempered steel rod and form block assembly with a radius of 5 mm and 90° angle (12, 23). The files were inserted into the form block so that the tip was just visible beyond the rod, and the rotational speed in this test was set at 240 rpm. Time to fracture was measured with a stopwatch to an accuracy of 0.1 seconds and the numbers of rotations calculated to the nearest full number. Pilot experiments had indicated that lubrication did not result in different mean cyclic fatigue scores but helped to reduce heat generated in the steel assembly. Therefore, a silicone-based lubricant was used (Motorex 622; Bucher, Langenthal, Switzerland).

Surface Evaluation

After determination of torsional and fatigue limits, file fragments were prepared for scanning electron microscopy. Care was taken to remove lubricant remnants; files were then mounted on stubs, sputter coated (Bal-Tec CSD 030; Bal-Tec, Balzers, Liechtenstein), and photographed at magnifications of $100\times$ and $400\times$ (model 1810, Amray, Cambridge, MA) to detect signs of corrosion.

Statistical Evaluation

Incidence of corrosion was compared by using chi-square tests. However, torque and fatigue data were normally distributed, and therefore statistical analysis for these parameters was done by parametric procedures. Two-way analysis of variance and Scheffé post hoc tests at $\alpha = 0.05$ (StatView 4.5; Abacus Concepts, Berkeley, CA) were used to evaluate the effect of immersion on torsional and fatigue resistance. The torque, angle, and number of rotations at fracture were dependent variables, whereas file type and immersion conditions were independent measures.

Results

After immersion in NaOCl, a total of 28 (12 ProFile, 16 RaCe, $p > 0.05$) files broke into two or more fragments because of obvious corrosion (Fig. 1), leaving 172 files for subsequent torque and fatigue testing. Files spontaneously separated significantly more frequently after

immersion in NaOCl at 60°C compared with 21°C (26/28, $p < 0.001$), whereas immersion duration had no effect on the separation incidence.

Torsional Resistance

Minimum and maximum torsional resistance were 1.12 and 17.1 Nm, respectively, whereas minimum and maximum angle at fracture were 98° and $1,043^{\circ}$, respectively. Figure 2 shows means and standard deviations of torque and angle at fracture in experimental and control groups. Overall, RaCe size 25 .04 files were less resistant to torsional load than ProFile size 25 .04 instruments ($p < 0.05$), whereas angles at fracture were not different. When subjected to NaOCl, mean fracture loads increased overall for both brands, but this difference was not significant (Fig. 2A). It resulted, however, in a significantly increased resistance to torsional load for RaCe files after 1 hour of immersion in NaOCl at room temperature ($p < 0.05$). Contact with heated NaOCl significantly reduced ($p < 0.001$) angle at fracture for RaCe files after 2 hours of immersion (Fig. 2B).

Fatigue Resistance

Minimum and maximum numbers of rotations to fracture were 7 and 629, respectively. Figure 2C shows means and standard deviations of rotations to fracture in experimental and control groups. Overall, ProFile size 25 .04 files were significantly more resistant to fatigue than RaCe size 25 .04 files ($p < 0.001$). Experimental conditions had a significant effect ($p < 0.001$) on fatigue resistance that affected both file types (Fig. 2C). The impact of NaOCl at room temperature for 1 hour was significant ($p < 0.05$) for ProFile only, whereas immersion in heated NaOCl reduced fatigue resistance for both brands by 40% or more ($p < 0.001$).

Surface Appearance

Scanning electron micrographs showed typical patterns of torsional and fatigue failure (data not shown). In some cases, pronounced deterioration was present (Fig. 1); the majority of the cases showed slight or no corrosion at all. Electropolished RaCe instruments had a subjectively smoother surface; however, there was no difference in corrosion patterns between the two file brands tested (Fig. 1A, B).

Discussion

This study assessed the effects of immersion of ProFile and RaCe NiTi rotary files in NaOCl solution and found a marked reduction of fatigue resistance. This finding is in agreement with a recent study on ProTaper (Dentsply Maillefer) files (20) but at variance with reports that resistance to fatigue of ProFiles was unaltered after immersion in NaOCl for 24 hours (24). Earlier, O'Hoy et al. (15) had shown that overnight incubation of various files brands with 1% NaOCl or Milton's solution did reduce resistance to fatigue. Possible explanations for these conflicting findings are different testing and immersion protocols.

In the current study, a testing platform was used that has been extensively validated for torque and fatigue testing, including simulated clinical instrument usage (12) and that was frequently calibrated. The attachment to test torsional loads conforms to the pertinent ISO norm (21) and did not show any signs of wear. However, the design used by O'Hoy et al. (15) to test torsional resistance employs forcing the rotating file into a small steel tube rather than stationary rotation and may therefore be subject to wear.

There was a slight increase in torsional resistance paralleled by a reduction in angle of fracture after immersion in NaOCl. This effect is variable and most likely clinically not relevant. A possible reason for this effect may be formation of a brittle surface layer during immersion of superelastic NiTi alloys (25).

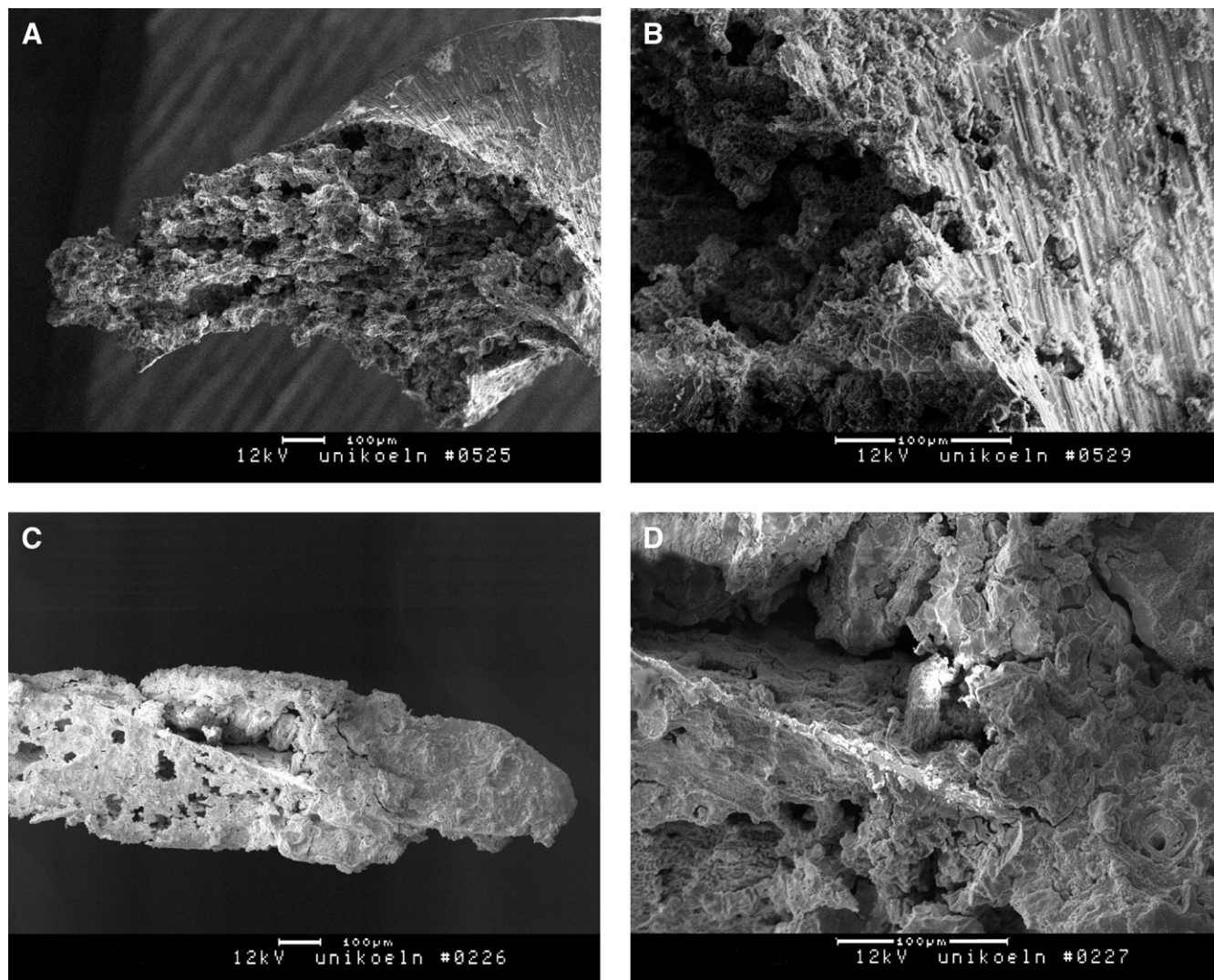


Figure 1. Scanning electron micrographs of corroded Ni-Ti files after immersion in 5.25% NaOCl for 2 hours at 60°C. Original magnification 100× (A, B) and 400× (C, D). (A, C) ProFile size 25 .04 taper. (B, D) RaCe size 25 .04 taper

It is noteworthy that, before immersion in NaOCl, ProFiles size 25 were more resistant to torque as well as fatigue than RaCe files of the same size. A possible explanation for this finding is the different cross section; however, the surface modification used in the production process of RaCe files (electropolishing) does not seem to be advantageous regarding fatigue failure under the conditions of the present study.

The procedure used in the present experiments to test fatigue resistance follows a design described by Haikel et al. (23), a modification of which incorporating a 45° or 60° instead of a 90° bend was also used by Castro Martins et al. (24) and Berutti et al. (20). Care was taken not to overheat or to wear out parts of the rod and from block assembly or the tested instrument because it had been described earlier that heat negatively affects fatigue resistance (26); therefore, it seems likely that testing conditions were relatively similar in the cited studies (20, 24) compared with the present experiment.

On the other hand, immersion conditions varied substantially, between 5 minutes (20) and 48 hours (27). Notably, the shortest reported immersion time was sufficient to cause reduced resistance to fatigue (20), whereas Haikel et al. (27) noticed no differences in torsional resistance after immersion in 2.5% NaOCl for 48 hours. It has been speculated that corrosion occurs more frequently and rapidly if not only

the working part of the instrument but also the shank part is inserted into the solution, enabling galvanic corrosion to take place (20, 24). In fact, obvious signs of corrosion similar to those seen after overnight immersion in 1% NaOCl or Milton's solution (15) were evident in the present study after complete immersion of the file.

Previous studies found fatigue fractures of new ProFiles size 25 .04 taper in a 45° curve after about 720 rotations (24); unused ProTaper F2 files separated in a 60° curve after a mean of 712 rotations. In the present study, ProFile size 25 .04 files fractured already after an average 490 rotations. However, a greater curve (90°) was used in the current study. Higher torque scores were recorded in the present study compared with earlier results (15, 27). However, smaller ProFile size 20 .04 were tested with friction at 300 rpm in one study (15), whereas a test using conditions stipulated by ISO3630-1 evaluated only hand files size #30 subjected to NaOCl (27).

Heating NaOCl solutions up to 60°C has been suggested to increase reactivity and antimicrobial and tissue-dissolving action (6, 8). However, the increase in reactivity may also enhance the corrosive potential of commercial NaOCl solutions (17) and adversely affect mechanical properties of NiTi rotaries. Such an effect of heating of NaOCl on NiTi file integrity was first described for ProTaper files by Berutti et al. (20). As

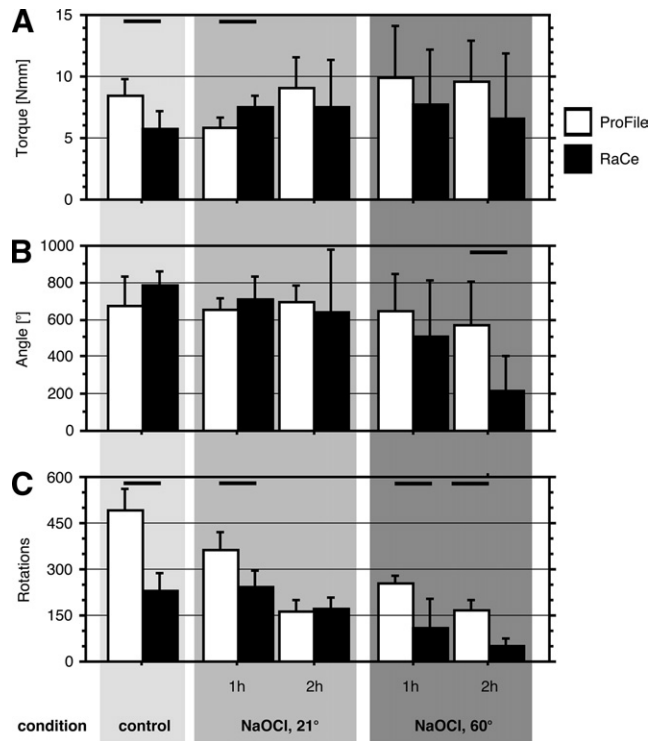


Figure 2. Diagrams presenting means (\pm standard deviation) of (A) torque, (B) angle, and (C) rotations at fracture. Experimental conditions (control, immersion in 5.25% NaOCl at 21°C and 60°C) are indicated by shading and results for ProFile and RaCe files are displayed by open and filled columns, respectively. Significant differences (analysis of variance with Scheffé tests) are indicated by horizontal bars.

expected, we found in the present study a reduction in fatigue resistance after immersion in NaOCl at 60°C, not only for machined ProFiles but also for electropolished RaCe rotaries.

The potential of corrosion, in particular in contact with NaOCl, of NiTi endodontic instruments (16) and orthodontic wires (28) has been reviewed recently (17). However, there is apparently no consensus whether or not corrosion presents a clinical problem. It needs to be pointed out that NaOCl is not only an indispensable endodontic irrigant (1, 2) but may be used to decontaminate files during reprocessing before sterilization (15, 19). Both applications would cause varying timeframes of contact, certainly beyond the 5 minutes of contact used, with subsequent deterioration of instrument surfaces, by Berutti et al. (20). On the other hand, it may not be feasible to clean instruments and remove adhering material without complete immersion in solution. Moreover, this procedure would not conform to suggested prion decontamination protocols (19). It is in fact questionable if current cleaning protocols regardless of the solutions used are capable of completely removing adherent particulate matter (19, 29).

We observed signs of gross corrosion in 28 of 160 NiTi files placed in NaOCl, 26 of which corroded in heated solution. This finding is likely a consequence of galvanic corrosion that may lead to very fast total destruction in few cases but only to minor areas of pitting in others. A potential mechanism for the development of a galvanic element is a breach of nickel or gold plating in the handle part (F. Aeby, personal communication, June 2006). In this type of corrosion, nobler areas are left behind to give the impression of a sponge-like structure (17). Because minor defects such as manufacturing marks may be initiation points for subsequent instrument fracture, it is conceivable that resis-

tance to cyclic fatigue is reduced by small and localized areas of pitting corrosion.

In contrast to previous findings (30), electropolishing RaCe files did not have a beneficial effect on resistance to corrosion under the present experimental conditions because a similar number of ProFile and RaCe instrument showed gross corrosion; furthermore, fatigue resistance of RaCe files was more impacted by contact with heated NaOCl solution than that of ProFiles.

In conclusion, there appears to be a risk of corrosion of NiTi rotary files in contact with NaOCl, especially when heated solution is used. Under the conditions of this in vitro study, both ProFile and RaCe rotaries showed a reduction in fatigue resistance but not in torsional strength. Visual inspection is recommended when reprocessing files with NaOCl to detect signs of gross corrosion. However, single use of NiTi rotaries may be indicated to reduce the danger of premature fatigue failure after contact with NaOCl.

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