Cyclic Fatigue Resistance of Heat-treated Nickel-titanium Instruments after Immersion in Sodium Hypochlorite and/or Sterilization

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Abstract

Introduction: The purpose of this study was to assess the effects of sodium hypochlorite (NaOCl) immersion and sterilization on the cyclic fatigue resistance of heat-treated nickel-titanium (NiTi) rotary instruments. **Methods:** Two hundred ten new 25/.06 Twisted Files (TFs; SybronEndo, Orange, CA) and Hyflex CM (Coltene Whaledent, Cuyahoga Falls, OH) files were divided into 7 groups (n = 15) for each brand. Group 1 (control group) included new instruments that were not immersed in NaOCl or subjected to autoclave sterilization. Groups 2 and 3 were composed of instruments dynamically immersed for 3 minutes in 5% NaOCl solution 1 and 3 times, respectively. Groups 4 and 5 consisted of instruments only autoclaved 1 and 3 times, respectively. Groups 6 and 7 recruited instruments that received a cycle of both immersion in NaOCl and sterilization 1 and 3 times, respectively. Instruments were subsequently subjected to a fatigue test. The surface morphology of fractured instruments was studied by field-emission scanning electron microscopy and x-ray energy-dispersive spectrometric (EDS) analyses. The means and standard deviations of the number of cycles to failure (NCF) were calculated and statistically analyzed using 2-way analysis of variance ($P < .05$). **Results:** Comparison among groups indicated no significant difference of NCF ($P > .05$) except for the groups of TFs sterilized 3 times without and with immersion in NaOCl ($P < .05$). HyFlex CM files exhibited higher cyclic fatigue resistance than TFs when files were sterilized 3 times, independently from immersion in NaOCl ($P < .05$). EDS analysis showed the presence of an oxide-rich layer on the Hyflex CM files' external surface. No morphologic or chemical differences were found between files of the same brand subjected to different treatments. **Conclusions:** Repeated cycles of sterilization did not influence the cyclic fatigue of NiTi files except for TFs, which showed a significant decrease of flexural resistance after 3 cycles of sterilization. Immersion in NaOCl did not reduce significantly the cyclic fatigue resistance of all heat-treated NiTi files tested. *(J Endod 2018;■:1–6)*

Key Words

Autoclave sterilization, controlled memory wire, cyclic fatigue, heat-treated files, sodium hypochlorite, R-phase

Significance

Immersion in sodium hypochlorite as well as autoclave sterilization could influence the cyclic fatigue resistance of NiTi instruments. It is important to know because these procedures influence the flexural fatigue of heat-treated NiTi files.

Root canal instruments manufactured from nickel-titanium (NiTi) alloy were introduced in 1988 to overcome the rigidity of stainless steel material (1, 2). However, there is a general perception that NiTi instruments have a high risk of fracture during their use (3), and manufacturers have continued their efforts to improve the properties and fracture resistance of NiTi instruments. Separation of NiTi files can be caused by torsional or flexural fatigue (cyclic fatigue) (4–6).

Many factors can influence the fatigue of NiTi instruments such as the type of kinematics (7) as well as raw materials and manufacturing processes (8). Resistance to cyclic fatigue of NiTi rotary instruments can be increased by improvements in the manufacturing process or the use of heat-treated alloys with superior mechanical properties (9).

The Twisted File (TF; SybronEndo, Orange, CA) is a NiTi rotary system manufactured with R-phase alloy using a twisting method. Its manufacturing process involves the transformation of a basic austenite NiTi wire into the R (premartensitic)-phase by a process of heating and cooling. After the twisted shape is achieved, a series of heating and cooling is said to convert the twisted R-phase wire back to the austenite crystalline structure, which becomes superelastic while stressed (10). This treatment would impart an increased cyclic fatigue resistance to the files (11).

Another development in the fabrication of endodontic NiTi instruments was the introduction of controlled memory wire (CM Wire; DS Dental, Johnson City, TN) as Hy-

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flex CM (Coltene Whaledent, Cuyahoga Falls, OH) files. These instruments have high resistance to cyclic fatigue and do not rebound to their original shape because of their alloy and specific manufacturing process (11, 12).

Because postmachining thermal treatment influences the properties of heat-treated alloys, the heat generated by sterilization procedures could influence the mechanical properties of endodontic instruments (9, 13). Sterilization of NiTi files must be ensured before clinical use except for the presterilized ones. Repetitive use of files under clinical conditions requires autoclave sterilization after every use. Also, prearranged sets of selected files may not be used during the same appointment. As a result, the unused rotary files are also subjected to multiple autoclave cycles (13, 14). Researchers reported that the additional "heat treatment" during autoclave sterilization might improve the flexibility of files, and the sterilization of files by using dry hot air and an autoclave would have a positive effect on the cyclic fatigue resistance (13, 14).

Although no effect of autoclave sterilization on Lightspeed NiTi rotary instruments (Discus Dental, Culver City, CA) has been reported, ProFile NiTi rotary files (Dentsply Maillefer, Ballaigues, Switzerland) have shown a higher mean cycle to failure when exposed to both dry heat and autoclave sterilization (14, 15). Similar findings of autoclave conditions either improving or degrading both the performance and physical properties of various marketed rotary NiTi systems have been reported (9, 14–17).

One additional factor potentially limiting the resistance of NiTi files to fatigue fracture is corrosion, which may occur in the presence of sodium hypochlorite (NaOCl) solution (18). NaOCl is used as an irrigant, and it selectively removes nickel from the instrument surface and causes micropitting (19), negatively affecting the physical and mechanical properties of NiTi files (20).

Even if the effects of NaOCl contact or autoclave sterilization on the cyclic fatigue of NiTi instruments have been separately investigated (14–18, 21), few studies analyzed the influence of both autoclave sterilization and NaOCl immersion on the cyclic fatigue of NiTi endodontic files (9, 22, 23). Moreover, no studies have investigated the influence of autoclave sterilization and immersion in NaOCl of instruments made by different heat-treated alloys such as the TF and Hyflex CM. Thus, the purpose of this study was to investigate the effect of both NaOCl contact and repeated autoclave cycles on the cyclic fatigue resistance of R-phase and CM Wire files.

Materials and Methods

Sample size estimation was calculated a priori with G*Power 3.1.9.2 software (Heinrich-Heine-Universität Düsseldorf, Düsseldorf, Germany). A total of 210 new 25/.06 TFs and Hyflex CM files were selected for the test. All instruments had been previously inspected using a measuring stereomicroscope (SZR-10; Optika, Ponteranica, Bergamo, Italy) for any signs of visible deformation. None were discarded.

Instruments all from the same production lot were randomly assigned into 7 groups (n = 15) for each brand. Group 1 (the control group) included new instruments that were not immersed in NaOCl or subjected to autoclave sterilization. Groups 2 and 3 were composed of instruments dynamically immersed for 3 minutes in 5% NaOCl solution (Niclor; OGNA Laboratory, Milan, Italy) at 37°C 1 and 3 times, respectively. All files were placed in small separate glass containers with the amount of NaOCl solution necessary to contact 16 mm of the instrument's length; dynamic immersion was allowed, activating the endodontic instruments with a 6:1 reduction handpiece (Sirona Dental Systems GmbH, Bensheim, Germany) powered by the VDW Silver Reciproc motor (VDW, Munich, Germany) at 500 rpm. Immediately after removal from the solutions, all files were rinsed with bidistilled water to neutralize the effect of NaOCl, dried, registered with an identification number, and stored in glass vials. Groups 4 and 5 consisted of instruments only autoclaved 1 and 3 times, respectively. The remaining groups (groups 6 and 7) recruited instruments that received a cycle of both immersion in NaOCl (as described previously) and sterilization 1 and 3 times, respectively.

The sterilized instruments were subjected to 1 or 3 cycles of autoclave sterilization (Sterilix Vacuum Plus; Reverberi, Barco, Italy), and each cycle was performed at a temperature of 134°C for 17 minutes (9). Each file was placed in a separate endodontic sponge and packaged singularly for sterilization in pouches. Instruments that underwent multiple autoclave cycles were allowed to cool to room temperature after sterilization. Sponges were removed from sterilization packaging and repackaged singularly in pouches before the subsequent autoclave cycles. In groups 4 and 5, no additional cleaning or surface treatment procedures were performed on the files before, during, or after sterilization. In groups 6 and 7, immersion in NaOCl was performed before autoclaving.

Instruments of all groups of each brand were then subjected to cyclic fatigue testing using an artificial stainless steel canal. It consists of a 36.8 mm × 25.4 mm × 9.5 mm metal block with a suitable artificial canal with a 60° angle of curvature and a 5-mm radius of curvature to the center of the 1.5-mm-wide canal. The radius was measured to the central axis of the curvature according to the method of Schneider (24). The center of the curvature was 5 mm from the tip of the instrument. The apparatus enabled the instrument to rotate freely within a stainless steel artificial canal at a constant pressure (7, 24, 25). The apparatus was connected to the same 6:1 reduction handpiece and motor used for dynamic immersion.

Cyclic fatigue tests were performed by rotating the instruments in continuous rotation at 500 rpm (as suggested by the manufacturers). Torque was set at the maximum level. The canals were covered with glass to prevent the instruments from slipping out (26). To reduce friction between the instrument and the metal canal walls, a special high-flow synthetic oil designed for lubrication of mechanical parts (Super Oil; Singer Co Ltd, Elizabethport, NJ) was applied.

All instruments were rotated until fracture occurred; timing was stopped as fracture was detected visually and/or audibly. To obviate human error, video recording was performed simultaneously, and the recordings were observed to cross-check the time of file separation.

The number of cycles to failure (NCF) for each instrument was calculated by multiplying the number of rotations per the effective seconds of continuous rotation required for fracture. The length of the fractured file tip was measured by using a digital microcaliper (Mitutoyo Italiana srl, Lainate, Milan, Italy).

Data were subjected to the Shapiro-Wilk test to characterize their normality and statistically analyzed using 2-way analyses of variance and the Tukey multiple comparison post hoc test to assess significant differences among groups ($P < .05$).

The broken fragments were analyzed using a field-emission scanning electron microscope (Nova NanoSEM 450; FEI, Eindhoven, the Netherlands) using 600× magnification to investigate the morphology of the fracture surface and 500× for the lateral views. This magnification was chosen to disclose the main feature morphology while maintaining a large field of view.

The chemical composition of the files' surface and eventual debris identified by imaging was obtained by a field-emission scanning electron microscope equipped with a Si-drift detector for energy-dispersive X-ray spectrometry (Quantax-200; Bruker, Berlin, Germany). Energy-dispersive X-ray spectrometric (EDS) spectra were collected from the middle region of each fractured specimen on 2000× magnified images.