



EFFECT OF SODIUM HYPOCHLORIDE AND EDTA GEL ON THE SURFACE TOPOGRAPHY OF TWISTED NICKEL-TITANIUM FILES USING AFM- AN *IN-VITRO* STUDY.

Dental Science

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ABSTRACT

Aim : To evaluate the effect of 3% NaOCl and 15% EDTA gel on the surface topography of Twisted file NiTi instruments using AFM analysis

Methodology : A total of 10 Twisted Files (Sybron Dental Specialties, Orange, CA, USA) .06#25, 23mm were divided into two groups: immersion in 3% NaOCl for 5 min and immersion in 15% EDTA gel for 5 min. Ten surface areas along 3- mm sections at the tip of the files (perfect squares of 2x2 mm) were analysed by AFM operating in contact mode under ambient conditions. Three-dimensional images (200 x 200 lines) were processed using Picoscan version 5.4 system software, and the root mean square value (RMS) of the scanned surface profiles were recorded. Data were analysed statistically.

Results : Three-dimensional AFM images of the surface of twisted files of , those immersed in NaOCl and EDTA gel, revealed topographic irregularities at the nanometric scale. RMS values of instruments treated with NaOCl and EDTA solutions were statistically higher than that of the new ones ($P < 0.05$).

Conclusion : Atomic force microscopy three-dimensional images and roughness values indicated that short-term contact between NaOCl and EDTA endodontic irrigants and twisted files instruments caused alterations on the surface of instruments.

KEYWORDS

INTRODUCTION

Nickel-titanium (NiTi) instruments have revolutionized the treatment aspects in endodontics. Despite many advantages of NiTi instruments, unexpected fractures can occur during clinical use.¹ Manufacturers have attempted various fabrication and annealing techniques to improve resistance to torsional fatigue.² However, mechanical grinding methods can potentially compromise NiTi wire grain structure and introduce imperfections into the NiTi crystalline lattice, which may negatively impact the physical properties of the NiTi wire.³

Recently, Gambarini *et al.*,⁴ described Twisted File (TF) (SybronEndo, Orange, CA) fabrication as transforming basic austenite NiTi wire into the R (premartensitic)-phase by a process of heating and cooling. Since R-phase NiTi wire is not amenable to grinding, only mechanical twisting can provide the desired configuration. After the twisted shape is achieved, a series of heating and cooling process is said to convert the twisted R-phase wire back to the austenite crystalline structure, which restores the super-elasticity of the file.

Repeated sterilization procedures plus contact between the NiTi instruments and the irrigating solutions, including sodium hypochlorite (NaOCl) and ethylenediaminetetraacetic acid (EDTA), can enhance their corrosion and deterioration which could, in turn, influence their mechanical properties and lead to undesirable fracture.⁵ Sodium hypochlorite (NaOCl) is the most common irrigant used in root canal treatment which often tends to discolour,¹³ and corrode surgical instruments.¹⁴ NiTi instruments come into contact with NaOCl during disinfection,¹⁵ or when the solution is present in the pulp chamber and root canal during instrumentation. Likewise, EDTA gel is most commonly used during chemo-mechanical debridement results in prolonged contact with Ni-Ti instruments.⁴⁴ Earlier studies have proved that irrigating solutions like EDTA and NaOCl have a deteriorating effect on the Ni-Ti files that have been manufactured by grinding process.¹⁶ In a recent study using AFM analysis on the surface of ProTaper NiTi files have shown to cause topographic irregularities when immersed in NaOCl and EDTA solutions.¹⁸ However, there is lack of evidence on the effect of these irrigants on files manufactured with a different alloy phase (R-phase). Also, no studies have been reported on the effect of EDTA gel on Ni-Ti files which is commonly used as a lubricant by clinicians.

Hence, the aim of our study was to evaluate the effect of 3% NaOCl and 15% EDTA gel on the surface topography of Twisted file rotary NiTi instruments using AFM analysis.

MATERIALS AND METHODS

A total of ten .06 #25 size, 23mm Twisted Files (Sybron Dental Specialties, Orange, CA, USA lot no.21096685, 121096685, 031021097) were divided into two groups: 3% NaOCl and 15% EDTA gel (n=5). Pre-exposure scan of all the files were recorded which served as the control. The new files were attached to a metal holder using rapid-drying cyanoacrylate glue. Each sample was placed on the AFM (Agilent technologies Picoscan 5100 LE, U.S.A.), and then 10 areas of the surface were analyzed on a 3-mm section at the tip of the files. The AFM images of the file samples were recorded using the contact mode operation under ambient conditions. Atomic force microscopy probes (silicon nitride) (curvature radius < 10 nm) mounted on cantilevers (250 μm), with a spring constant of 0.1 N m), were used. Scanned areas were perfect squares (2x2 μm). Three dimensional images (200X200 lines) were processed using Picoscan version 5.4 system software.

Following this, the Ni-Ti files were totally immersed in 3% NaOCl (Vensons, Bangalore, India batch no. 13100) at Ph 11 or 15% EDTA Gel (Dentsply Mallifer USA LOT NO. 100914) at Ph 7 for 5 min, respectively. The files were then rinsed using distilled water to remove traces of the irrigants on the surface and left to dry in normal atmospheric conditions prior to post-exposure AFM scan.

The root mean square of the scanned surface profiles was recorded. These parameters indicated changes in vertical surface topography, and an increase in RMS values meant alterations of NiTi instruments surface caused by the irrigants. Statistical analysis of the data was performed using SPSS Software version 15.0. The pre and post-immersion RMS values within the groups were analyzed statistically by the Wilcoxon signed ranked test and Mann Whitney U test was used to compare the mean difference RMS values (post immersion RMS – Pre-immersion RMS) between the groups. The level of significance was set at $P < 0.05$

RESULTS

Three dimensional surface roughness of the new files and those

immersed in NaOCl solution and EDTA gel showed topographical changes at the nanometric scale. (Fig.3) The RMS values of the files are given in Table 1. There was a statistically significant difference in the pre- and post-immersion RMS values of the Ni-Ti instruments for both 3% NaOCl and 15% EDTA gel. Hence, the increase in RMS values was dependent on the time and irrigating solution used. (P < 0.05)

Comparison of mean difference RMS values using Mann Whitney U test showed 15% EDTA gel to be statistically superior to 3% NaOCl. In other words NaOCl was more corrosive with respect to the surface topography of the Ni-Ti files than 15% EDTA gel. (Table 2)

DISCUSSION

Despite several improvements in the manufacture of Ni-Ti files, instrument separation is still a major concern when using NiTi rotary files.^{20,22,24} Cyclic fatigue failure is reported to occur unexpectedly without any sign of previous permanent deformation.^{25,26} This occurs because of the alternating tension/compression cycles that instruments are subjected to when flexed in the region of maximum curvature of the canal.^{27,28,29} Many variables such as the operational speed³¹, the effects of the irrigant's action,¹² the metal surface treatments,³⁰ and the metallurgic characterization of the NiTi alloys that could possibly influence the fatigue resistance of NiTi rotary files have been investigated.³¹

During chemo-mechanical debridement, rotary Ni-Ti files are exposed to irrigants.³² Sodium hypochlorite and EDTA are commonly used. Sodium hypochlorite in endodontics is used in various concentrations (1%-5.25%). In the present study 3% Sodium hypochlorite (Vensons, Bangalore, India) was used at a Ph of 11. A five minute contact time of Sodium hypochlorite with the total immersion of the instrument was selected to mimic the realistic times during clinical practice¹⁸. EDTA is commonly used as a 17% solution or gel form. In the gel form (Dentsply Mallifer USA) it is available in the concentration of 15% at a Ph of 7. Earlier studies have used EDTA in a solution form on Protaper Ni-Ti files.¹⁶ However, EDTA liquid may often be used as a final irrigant which virtually eliminates the contact with Ni-Ti files. Moreover, EDTA in gel form is a recommended lubricant to be used with Ni-Ti files. Hence, in the present study EDTA gel was used as it may have a prolonged contact time on the surface of Ni-Ti files. A five minute contact time of EDTA gel with the instrument was selected so as to emphasize its effect whilst remaining within realistic times for clinical practice.

NiTi files may be susceptible to the highly corrosive effects of the popular irrigant NaOCl^{10,38,39,6,40}. Corrosion may also occur during chemical sterilization with NaOCl.^{11,41} An investigation by Berutti *et al.*,⁸ showed a decrease in cyclic fatigue resistance when Protaper NiTi files were completely immersed in 5% NaOCl for five minutes. On the other hand, few other studies have reported that NaOCl and EDTA exposure do not affect the cyclic fatigue of the NiTi instruments.^{6,10,9,42}

Earlier studies on the effect of NaOCl on rotary Ni-Ti instruments manufactured by grinding process following clinical use concluded that there was a significant decrease in resistance to cyclic fatigue.^{16,17,39} However, TF files employ a different alloy phase of Ni-Ti technology and the instrument is developed by twisting as compared to grinding. Size 25, 0.06 taper TF were used in the present study as it is similar in diameter to ProTaper F2 Ni-Ti files that were used in earlier studies. Additionally, these are the initial instruments that were used to complete the preparation of the apical stop in their respective rotary systems. Furthermore, these instruments are used along the entire length of the root canal and, therefore, undergo both torsional and flexural loads. Ametrano *et al.*, observed maximum corrosion in Protaper finishing files compared to shaping files.¹⁸

The AFM is a now well established and documented technique to provide qualitative and quantitative information about the topography of a wide variety of materials.³³⁻³⁷ SEM and AFM provide resolution of structure down to the nanometric scale. However, the image formation mechanisms are quite different, resulting in different types of information. SEM uses an electron beam operated under vacuum to give a two-dimensional 'photographic' image of the samples; but cannot directly provide quantitative data regarding the topography.^{36,37} In contrast, the AFM technique reconstructs, in real time, the three-dimensional image of the sample topography on a computer screen and also records the experimental data in digital form as sets of z (vertical axis), x (horizontal axis), and y (transversal axis) values. These sets can be analyzed with dedicated digital software to give all the data pertaining to the examined surface in quantitative form at nanometric scale.³³⁻³⁷

Both irrigants caused significant deterioration of instrument surfaces resulting in an increase in RMS values compared to new instruments. The present findings on NaOCl are in line with the previous studies, whereas they contradict other recent investigations which reported no effects caused by NaOCl and EDTA on NiTi instruments with different surface treatments.^{7,11,43} Darabara *et al.*,¹¹ used the cyclic potentiodynamic polarization method to evaluate the pitting and crevice corrosion characteristics of NiTi instruments exposed to 17% EDTA and 5.25% NaOCl solutions. They demonstrated high corrosion resistance of the instruments to both irrigating substances. Bonaccorso *et al.*,⁷ confirmed that long-term EDTA contact did not alter the surface structure of files. However, the data from the previous studies were based on different contact times between files and irrigants and on SEM analysis instead of AFM, which might explain the differences between the results.

In the present study the results showed that exposure of five minutes to NaOCl and EDTA caused corrosion and resulted in alteration of surface topography of twisted rotary Ni-Ti files (figure 3). The newer manufacturing method of twisting did not prevent surface deterioration of the TF. The surface deterioration was significantly higher among files immersed in NaOCl compared to EDTA. In a similar study, Nova *et al.*, found that with total file immersion a galvanic coupling is created between the instrument's shank and the nickel-titanium working end when it is placed in an electrolytic solution (NaOCl). This electro-chemical reaction creates an environment which may lead to corrosion. This was in particular observed with files like Protaper which have different metal plating on the shank.³⁹

There has been compelling evidence that surface roughness / micro cracks are directly related to fracture of NiTi files. This surface roughness present on new files coupled with corrosive nature of the irrigants could predispose the NiTi files to early fracture. Hence, it would be prudent to restrict the usage of these files to a single use; which has been emphasized in previous studies.^{19,23} However, further studies are needed to explain the mechanism and type of corrosion that occurs in twisted files and also to compare the effect of other endodontic irrigants on surfaces of various rotary instruments and its effect on cyclic fatigue during clinical performance.

CONCLUSION

Within the limitations of the present study, it was concluded that on short term contact, both 3% NaOCl and 15% EDTA gel deteriorated the surface of twisted NiTi files. Among the two groups, 3% NaOCl was more corrosive than 15% EDTA gel.

Table 1. Mean RMS values of pre and post immersion values for 3% NaOCl and 15% EDTA gel

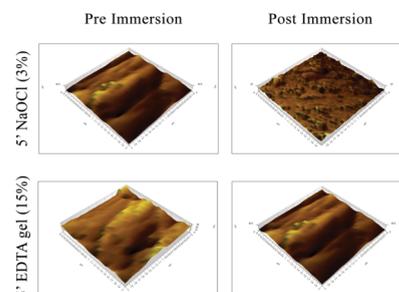
Groups	Numbers	Mean ±SD		P value
		Pre- Exposure	Post-Exposure	
3% NaOCl	5	2.56±.42	4.41±.48	0.043*
15% EDTA	5	3.63±.85	4.33±.99	0.043*

* P<0.05

Table 2. Mean Difference RMS values for 3% NaOCl and 15% EDTA gel

	Numbers	Minimum	Maximum	Mean difference	SD	P value
3% NaOCl	5	0.85	2.86	1.85	0.73	0.016*
15% EDTA	5	0.50	0.91	0.70	0.18	

* P<0.05



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