

Qualitative Assessment of the Surface Topographic Changes of XP-endo Shaper and TruNatomy files after exposure to Sodium Hypochlorite and Ethylenediaminetetraacetic Acid

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ABSTRACT

Objective: TruNatomy and XP-endo Shaper are recently introduced file systems showing increased fatigue resistance rate. The present study aims to evaluate the surface topographic changes and nickel (Ni) and titanium (Ti) elemental loss of XP-endo Shaper (XPS) and TruNatomy (TN) files on exposure to conventionally used root canal irrigants; [5.25% sodium hypochlorite (NaOCl) and 17% ethylenediaminetetraacetic acid (EDTA)] at a 10 minute time frame using atomic force microscopy (AFM) and energy dispersion X-ray spectroscopy (EDX) analytical techniques.

Methods: Twelve samples for each of XPS (30/.04 taper) and TN (26/.04 taper; prime) instruments were dynamically exposed to 5.25% NaOCl, 17% EDTA separately for 10 minutes and in combination of 5.25% NaOCl (8 minutes)+17% EDTA (2 minutes) for a total of 10 minutes. Post exposure, the files were subjugated to AFM and EDX analysis. Independent t test and one-way ANOVA were used for statistical analysis, and the level of significance was set at 0.05.

Results: XPS and TN showed a significant increase of surface roughness (Ra) and roughness mean square (RMS) on exposure to various irrigants ($P < 0.05$) using AFM analysis. Increased overall roughness was observed with TN in comparison to XPS ($P < 0.05$). Elements Ni and Ti loss was found in both XPS and TN files using EDX analysis. Both files exhibited Ni and Ti loss with the loss of Ni content higher for TN after exposure to 17% EDTA. Loss of Ti was seen for both files on exposure to a combination of 5.25% NaOCl+17% EDTA.

Conclusion: After exposure to root canal irrigants, the surface roughness was lesser in XPS compared to TN files. 17% EDTA caused significantly higher surface roughness in both file systems when compared to 5.25% NaOCl. TN exhibited overall higher elemental (Ni and Ti) loss on exposure to 17% EDTA and 5.25% NaOCl+17% EDTA in comparison to XPS files.

Keywords: Atomic force microscopy, EDTA, endodontics, root canal preparation, sodium hypochlorite

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HIGHLIGHTS

- XP-endo Shaper showed an overall less surface roughness compared to TruNatomy when exposed to various commonly used root canal irrigants.
- XP-endo Shaper showed a lower net loss of mineral content compared to TruNatomy when exposed to various commonly used root canal irrigants.
- 17% EDTA caused higher surface roughness for both XP-endo Shaper and TruNatomy compared to 5.25% NaOCl and consecutive exposure of 5.25% NaOCl followed by 17% EDTA.
- Consecutive exposure to 5.25% NaOCl followed by 17% EDTA showed a resultant decrease in surface roughness for both XP-endo Shaper and TruNatomy in comparison to individual exposure to 5.25% NaOCl or 17% EDTA respectively.
- Higher loss of 'Ni' content was seen when both files were exposed to only 5.25% NaOCl or 17% EDTA. A higher loss of 'Ti' content was seen for both files when exposed to combination of 5.25% NaOCl and 17% EDTA.

INTRODUCTION

Mechanical instrumentation of the root canal system using nickel titanium (NiTi) files has gained popularity in recent years because of its resiliency, shape memory and superelasticity properties (1). The evolution of NiTi metallurgy in recent years have allowed manufacturers to develop variations in the structure of the endodontic files allowing for better resistance to instrument separation which is caused mainly due to cyclic or torsional fatigue (2). During the process of mechanical preparation of the canals, NiTi files when used in conjugation with conventional root canal irrigants is shown to effectively reduce the microbial load and aid in removal of smear layer (3, 4). Root canal irrigants such as sodium hypochlorite (NaOCl) and ethylenedi-

aminetetraacetic acid (EDTA) have been recommended to be used in combination for effective removal of the inorganic and organic components of the smear layer which may harbour microbial irritants (5).

Root canal irrigants are in frequent contact with the surface of endodontic files during the instrumentation process and has an effect on the surface of the files causing deleterious surface topographic changes, which may influence the incidence of fracture of the instrument (6). In an event of constant exposure of files to root canal irrigants, it has been reported that the flexural and torsional fatigue strength of files is greatly reduced (7), and can cause instrument separation (8).

Minimally invasive endodontics concept has shown to preserve the integral tooth structure, reducing the incidence of crack formation and preventing tooth fracture to occur by preserving the peri-cervical dentine (9). XP-endo Shaper is a newer generation rotary file system with snake-like pattern observed in its austenitic phase used for the 3 dimensional disinfection of the root canal system (10, 11). Recent evidence supports the use of these files for root canal preparation in conservative access cavities (12, 13). XP-endo Shaper is a single file system made with a proprietary 'MaxWire' technology showing to have increased flexibility and fatigue resistance (14). Another recently introduced system, TruNatomy is a special heat-treated file with a slim wire design and off-centered cross section. Recently, endodontic literature has shown increased fatigue resistance of these files (15, 16) with no studies published to date indicating the surface changes of these files on exposure to root canal irrigants which may influence the fracture incidence rate of the instrument. In regard to this, the present aim of our study was to evaluate the surface topographic changes and elemental loss (Ni and Ti) of XP-endo Shaper and TruNatomy files on exposure to conventionally used root canal irrigants at certain time intervals using atomic force microscopy (AFM) and energy dispersive X-ray spectroscopy (EDX) analytical techniques. The null hypothesis for the present study was considered that surface of endodontic files was not affected on exposure to root canal irrigants.

MATERIALS AND METHODS

Sample size calculation

The sample size estimation was based on the previous study by Cai et al. (17). Power calculation was done using G*Power 3.1 software for Windows (Henrick Heine-Universität, Düsseldorf, Germany). A total of 24 samples were taken with 12 per group indicated as the ideal sample size required for observing significant differences. The samples were again divided into 4 subgroups with 3 per group in regard to exposure to different root canal irrigants. ($d=1.26$; $\alpha=0.05$; $1-\beta=0.9$)

Study design and sample preparation

The study was conducted after the approval of the ethical committee of the university with the approval number of SRB/SDC/ENDO-1805/20/01 dated 21/07/2020. The study design was conducted according to the modification of the design followed in previously published studies (17, 18). Two opera-

tors were involved in the present study with the first operator being blinded about the type of irrigant used and exposed the NiTi instruments to the root canal irrigants and the second operator was blinded about the type of NiTi instrument and subjected the irrigant exposed instruments to AFM and EDX analysis.

Twelve files of XP-endo Shaper (XPS; FKG Dentaire SA, La Chaux-de-Fonds, Switzerland) of tip diameter size 25, a taper of 0.04 and length of 25 mm and twelve files of TruNatomy Prime (TN; Dentsply Sirona, Ballaigues, Switzerland) of size 26, a taper of 0.04 and length of 25 mm were selected to assess the surface topographic changes after exposure to 17% ethylenediaminetetraacetic acid (EDTA) (Meta – Biomed Co. Ltd, Chungcheongbuk-do, Korea) and 5.25% sodium hypochlorite (NaOCl) (Prime Dental Products Pvt Ltd, Thane, India). The files were taken from a sealed box and both TruNatomy and XP-endo Shaper files were again divided into subgroups and separately exposed to the selected endodontic irrigants at different time intervals as follows;

- Group A: On exposure to 5.25% NaOCl for 10 minutes
- Group B: On exposure to 17% EDTA for 10 minutes
- Group C: On exposure to combination of 5.25% NaOCl (8 minutes) and 17% EDTA (2 minutes)
- Group D: No exposure to any irrigant (Control)

The files on exposure to different root canal irrigants were exposed dynamically in a small glass container with sufficient contact with the surface of endodontic files and not the shaft of the file in order to prevent galvanic corrosion between the instrument and handle. The temperature of the intracanal irrigants was maintained in a water bath at a controlled temperature ($35\pm 1.0^{\circ}\text{C}$) to better simulate intracanal temperature. The files were attached to an endomotor (X-Smart Plus, Dentsply Maillefer, Ballaigues, Switzerland), for TruNatomy files the files were immersed in the irrigants and subjected to a continuous rotation at speed 500 RPM with torque of 1.5 Ncm^2 and for the XP-endo Shaper files were exposed to the irrigants in a similar fashion with settings of continuous rotation at speed 800 RPM with a torque setting of 1 Ncm^2 . Both settings were selected as recommended by the manufacturers. Immediately after exposing the files to set amount of time to all irrigants, they were washed with distilled water to neutralize the effect of the irrigants and dried. Followed by this, the endodontic files were then subjected to atomic force microscopy (AFM) and energy dispersive X-ray spectroscopy (EDX) analysis.

1) Atomic force microscopy

The NiTi files were placed on a glass base and fixed using a rapid setting cyanoacrylate glue. The AFM images were recorded in contact mode operation under ambient conditions using a XE7 Park System (Park Systems, Suwon, South Korea). A total of nine squares of 2×2 , 5×5 and 10×10 micrometer were examined on every sample, which was then unitized to 1×1 micrometer for statistical analysis. 3mm of the file from the tip was taken for analysis. The roughness average

(Ra), root mean square (RMS) and mean height of roughness profile elements (Rc) of the scanned profiles were then recorded. The three-dimensional images were processed using the Picoimage software (Keysight Technologies, Yishun Ave, Singapore).

2) Energy dispersive X-ray spectroscopy

The instrument used for evaluating the energy dispersive X-ray spectroscopy (EDX) was using FEA Company of USA (S.E.A) PTE LTD. EDX identifies the chemical elements by X-ray mapping the spatial distribution of these elements and a spectrum of energy demonstrates the relative number of chemical elements present, with a penetration power of 1 μm of the electron beam in order to determine qualitatively and quantitatively the elements present in the sample. The weight percentage of the elements present on the surface of the studied samples post exposure to various irrigants were evaluated.

Statistical analysis

The data was analysed using SPSS version 20 (IBM Corp, Armonk, USA). The normality tests, Kolmogorov-Smirnov and Shapiro-Wilks showed a normal distribution. To compare the topographic changes between the files post exposure to the irrigants, Independent t test was used. For comparison of the topographic changes of the files post exposure to the irrigants one-way ANOVA was used. To conduct multiple group comparison, post hoc tukey's test was applied. A p value of 0.05 was taken as statistical significance level.

RESULTS

The surface morphological changes of the selected files were examined post exposure to various irrigants. For the surface

roughness changes, the surface roughness area (Ra), root mean square (RMS), and roughness profile elements (RPE) were evaluated using atomic force microscopy (AFM) imaging and the surface elemental composition post exposure was done using energy dispersion X-ray spectroscopy (EDX).

Overall surface roughness, surface mean square and roughness profile elements were found to be significantly higher for TruNatomy files compared to XP-endo Shaper on exposure to 5.25% NaOCl ($P<0.05$), 17% EDTA ($P<0.05$), 5.25% NaOCl+17% EDTA ($P<0.05$) (Table 1).

For TruNatomy files, on pre-exposure to root canal irrigants exhibited lesser surface roughness when compared to post exposure (Table 2), The surface roughness was found to be significant higher on exposure to 17% EDTA ($P<0.001$) in comparison to 5.25% NaOCl ($P<0.001$), combined exposure to 5.25% NaOCl and 17% EDTA ($P<0.001$) yielded a significantly higher roughness in contrast to the control group without treatment (Fig. 1, Table 2). Surface elemental composition analysis using EDX yielded a higher loss of Ni content in 17% EDTA and 5.25% NaOCl and higher loss of Ti content in 5.25% NaOCl+17% EDTA (Fig. 2).

For XP-endo Shaper, the surface roughness was found to be as follows; 17% EDTA>5.25% NaOCl+17% EDTA>5.25% NaOCl>Control group ($P<0.001$) (Table 3, Fig. 3). Surface elemental analysis using EDX yielded a higher loss of Ni content in 17% EDTA and 5.25% NaOCl but was less in comparison to TruNatomy files group. Loss of Ti content was observed highest in 5.25% NaOCl+17% EDTA group and 17% EDTA, and overall considered to be lower in comparison to TruNatomy group.

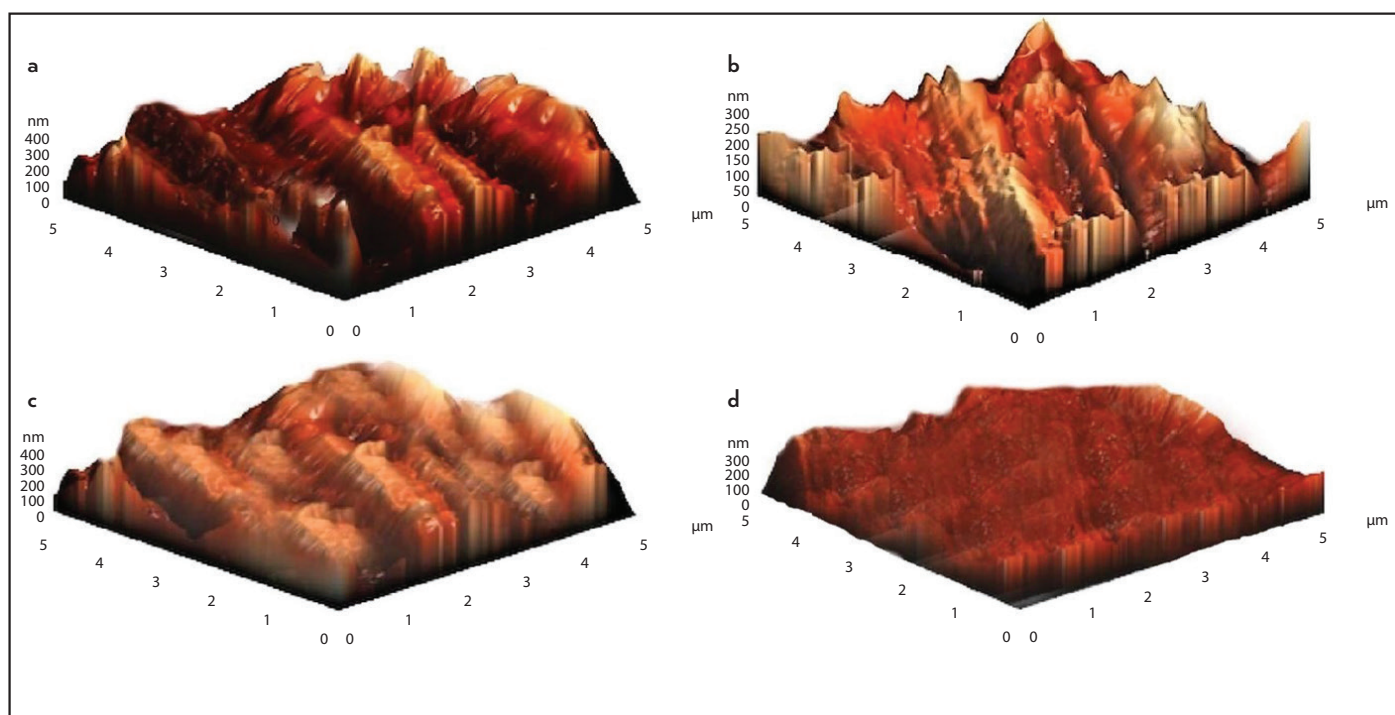


Figure 1. Atomic Force Microscopy imaging of TruNatomy files on exposure to various irrigants (a) On exposure to 5.25% NaOCl for 10 minutes, (b) On exposure to 17% EDTA for 10 minutes, (c) On exposure to combination of 5.25% NaOCl for 8 minutes and 17% EDTA for 2 minutes, (d) No exposure to any irrigant

TABLE 1. Overall effect of the surface roughness, root mean square, roughness profile elements of both files on exposure to different root canal irrigants

Root canal irrigant	Surface roughness parameters	XP-endo Shaper (Mean±SD)	TruNatomy (Mean±SD)	Mean difference	t value	P value
5.25% NaOCl	Roughness Area (Ra)	4.58±0.46	6.09±0.63	-1.51000	-3.322	0.02
	Root Mean Square (RMS)	3.71±0.41	7.25±0.29	-3.54000	-11.990	0.01
	Roughness Profile Elements (RPE)	3.14±0.19	7.99±0.46	-4.85000	-16.616	0.01
17% EDTA	Roughness Area (Ra)	6.64±0.53	9.56±0.47	-2.92	-7.041	0.02
	Root Mean Square (RMS)	6.94±0.16	11.25±0.25	-4.31	-24.788	0.01
	Roughness Profile Elements (RPE)	5.29±0.26	10.11±0.12	-4.82	-28.943	0.01
5.25% NaOCl+17% EDTA	Roughness Area (Ra)	5.21±0.33	8.51±0.54	-3.30	-8.891	0.01
	Root Mean Square (RMS)	3.24±0.22	8.24±0.11	-5.00	-34.233	0.01
	Roughness Profile Elements (RPE)	4.85±0.29	8.56±0.31	-3.71	-15.079	0.01
Control	Roughness Area (Ra)	2.05±0.08	5.08±0.46	-3.03	-11.062	0.01
	Root Mean Square (RMS)	2.24±0.11	6.12±0.01	-3.87667	-57.933	0.01
	Roughness Profile Elements (RPE)	3.17±0.17	6.89±0.29	-3.72000	-18.918	0.01

P value less than 0.05 was considered as significant

TABLE 2. Surface roughness, root mean square, roughness profile elements of TruNatomy files on exposure to various root canal irrigants

Surface roughness parameters	Root canal irrigants	Mean±SD	F value	P value
Surface Roughness Area (nm)	5.25% NaOCl	6.09±0.63	15.63	<0.001*
	17% EDTA	9.56±0.47		
	5.25% NaOCl+17% EDTA	8.51±0.54		
	Control	5.08±0.46		
Root Mean Square (nm)	5.25% NaOCl	7.25±0.29	12.1	<0.001*
	17% EDTA	11.25±0.25		
	5.25% NaOCl+17% EDTA	8.24±0.11		
	Control	6.12±0.01		
Roughness Profile Elements (nm)	5.25% NaOCl	7.99±0.46	4.57	<0.01*
	17% EDTA	10.11±0.12		
	5.25% NaOCl+17% EDTA	8.56±0.31		
	Control	6.89±0.29		

All the roughness parameters showed significant difference (P<0.05) on exposure to different root canal irrigants

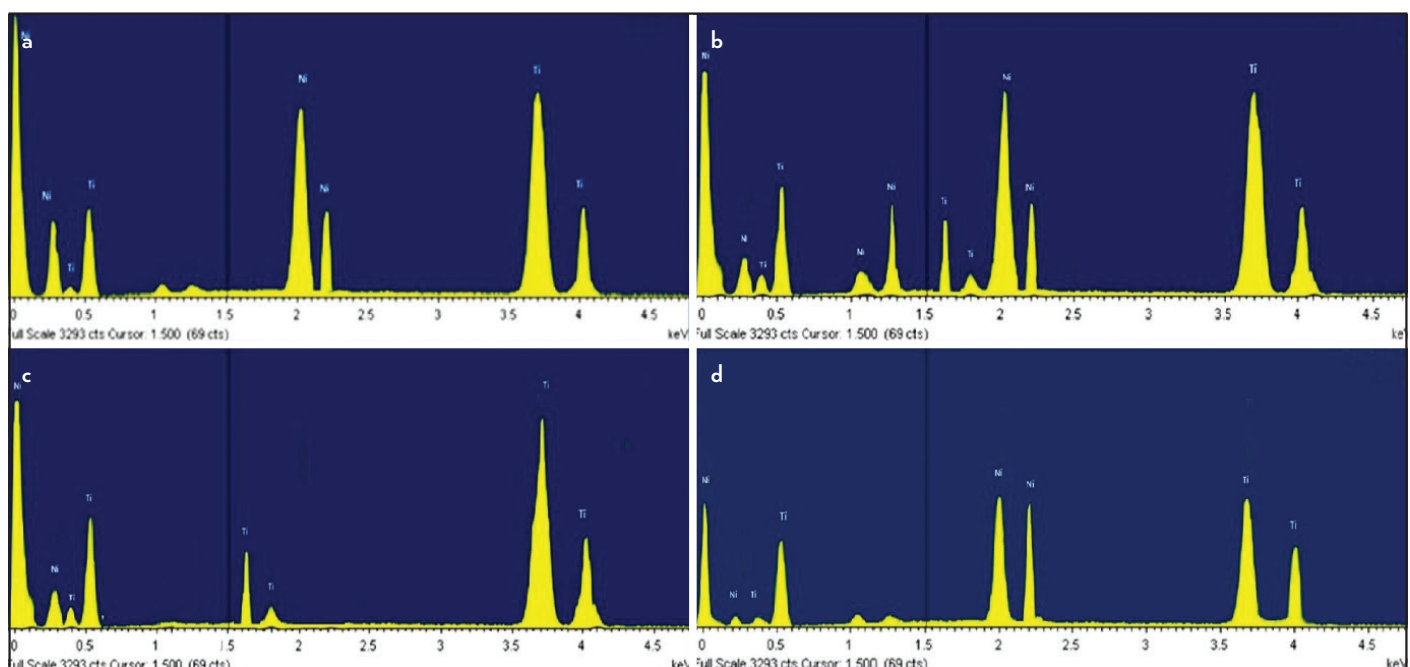


Figure 2. Surface elemental composition loss analysis of TruNatomy files on exposure to different irrigants (a) On exposure to 5.25% NaOCl, (b) On exposure to 17% EDTA, (c) On exposure to 5.25% NaOCl for 8 minutes and 17% EDTA for 2 minutes, (d) No exposure to any group

TABLE 3. Surface roughness, root mean square, roughness profile elements of XP-endo Shaper files on exposure to various root canal irrigants

Surface roughness parameters	Root canal irrigants	Mean±SD	F value	P value
Surface Roughness Area (nm)	5.25% NaOCl	4.58±0.46	7.95	<0.01*
	17% EDTA	6.64±0.53		
	5.25% NaOCl+17% EDTA	5.21±0.53		
	Control	2.05±0.08		
Root Mean Square (nm)	5.25% NaOCl	3.71±0.41	2.58	<0.05
	17% EDTA	6.94±0.16		
	5.25% NaOCl+17% EDTA	3.24±0.22		
	Control	2.24±0.11		
Roughness Profile Elements (nm)	5.25% NaOCl	3.14±0.19	3.05	<0.05
	17% EDTA	5.29±0.26		
	5.25% NaOCl+17% EDTA	4.85±0.29		
	Control	3.17±0.17		

All the roughness parameters showed significant difference (P<0.05) on exposure to different root canal irrigants

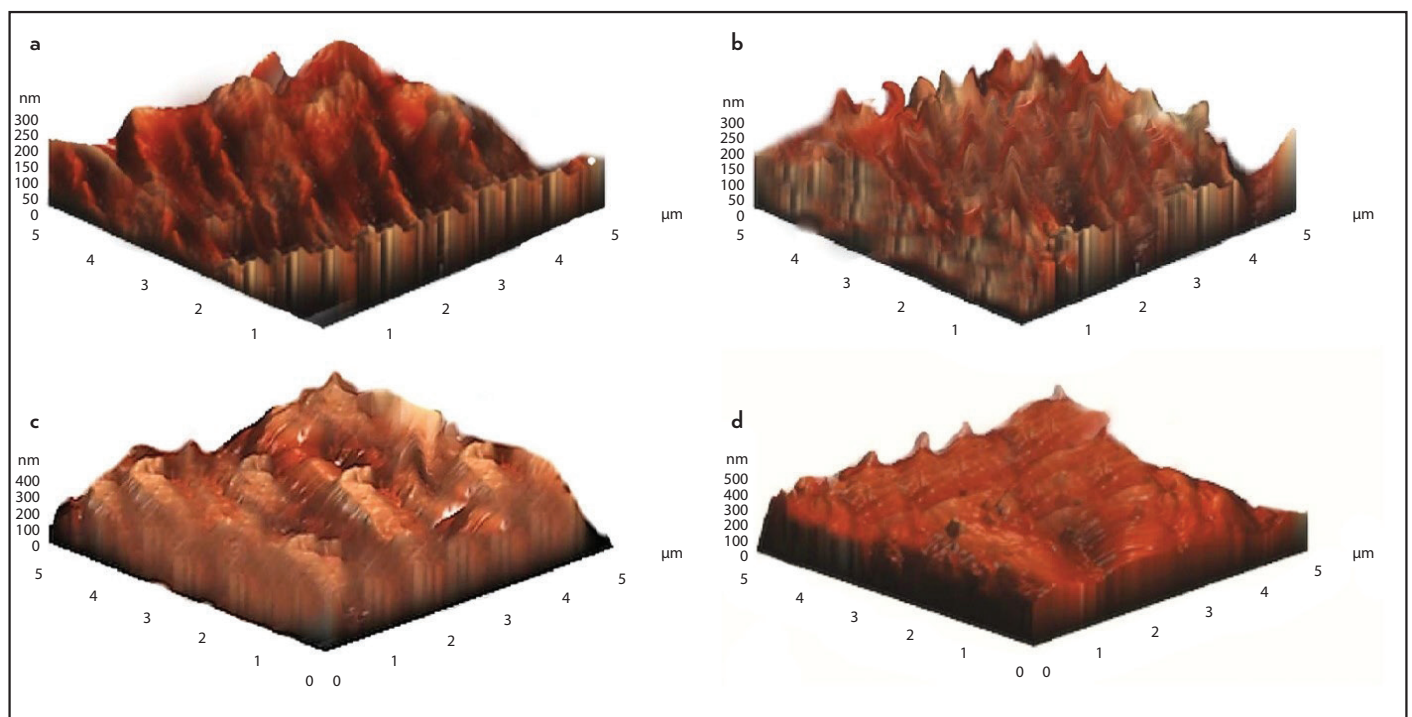


Figure 3. Atomic Force Microscopy imaging of XP-endo Shaper files on exposure to various irrigants (a) On exposure to 5.25% NaOCl for 10 minutes, (b) On exposure to 17% EDTA for 10 minutes, (c) On exposure to combination of 5.25% NaOCl for 8 minutes and 17% EDTA for 2 minutes, (d) No exposure to any irrigant

DISCUSSION

The purpose of the present study was to assess the surface topographic changes on exposure to commonly used root canal irrigants such as 5.25% NaOCl and 17% EDTA for a total time period of 10 minutes. The null hypothesis of the present study was rejected since changes to the files surface were observed after exposure to root canal irrigants. The evaluation of the surface topographic changes for the present study was done using atomic force microscopy (AFM) which is considered a useful microscopy technique due to its scope to qualitatively evaluate surface details down to the atomic level in a three-dimensional reconstruction (19) without any special treatment in comparison to other imaging methods. Further evaluation using energy dispersive X-ray spectroscopy (EDX) was done to evaluate the elemental composition loss of Ni and Ti after exposure to

different irrigants. EDX is considered as an elemental analytic technique which can quantitatively assess the analytical composition of elements in a sample (20). In this study, both XP-endo Shaper and TruNatomy files showed some degree of elemental loss on exposure to the selected irrigants. Exposure to 17% EDTA showed highest loss of Ni content in both the files used in this study. This result could be due to the extended contact time of the files potentially having an erosive effect on the surface of files. EDTA has shown to have a erosive effect on metal surfaces (21). The same results were not emulated in combination use of 5.25% NaOCl+17% EDTA where loss of Ni content was least compared to all groups but the loss of Ti content was seen to be highest in comparison to all other test groups.

NaOCl and EDTA are known to have properties such as anti-bacterial action, tissue dissolving property, smear layer re-

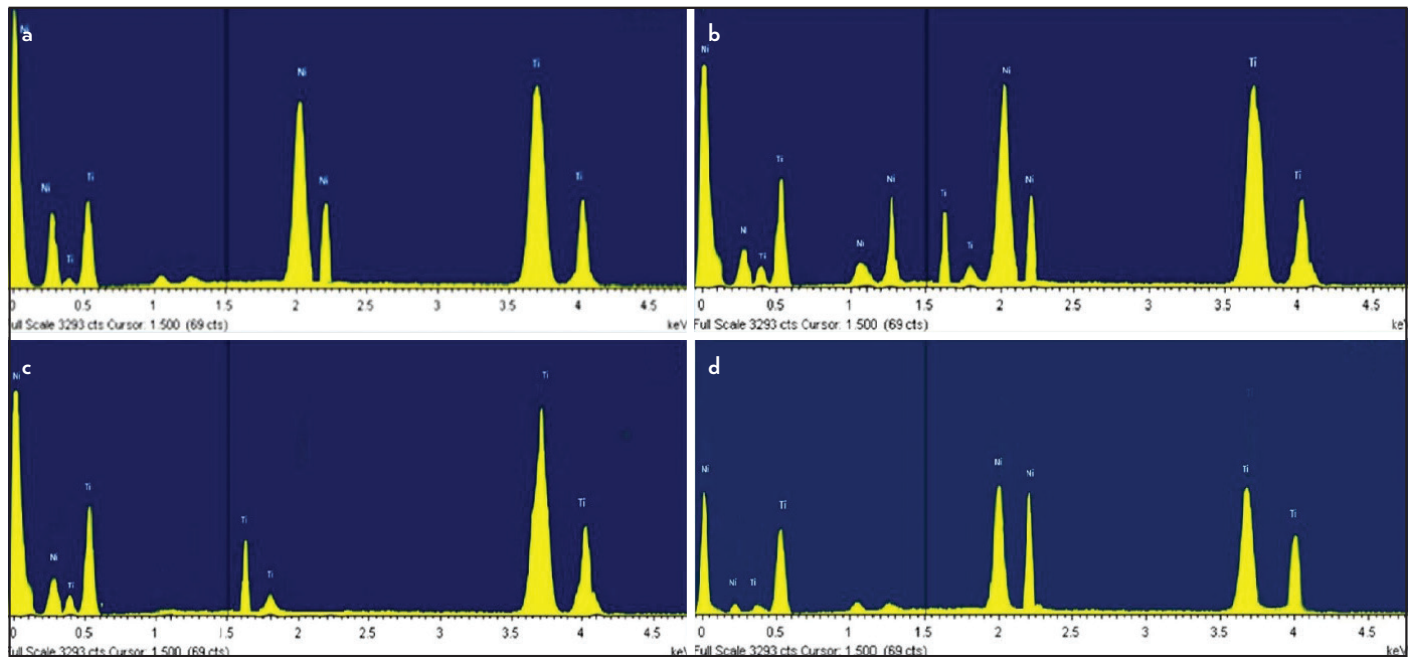


Figure 4. Surface elemental composition loss analysis of XP-endo Shaper on exposure to different irrigants (a) On exposure to 5.25% NaOCl, (b) On exposure to 17% EDTA, (c) On exposure to 5.25% NaOCl for 8 minutes and 17% EDTA for 2 minutes, (d) No exposure to any group

moval and ability to chelate the dentine which are used in combination and is recommended by American Association of Endodontists (22, 23). The usage of 5.25% NaOCl is shown to be significantly effective against microbes in comparison to lower concentration (23). 17% EDTA as a chelating agent has shown to be effective to eradicate remaining microbes, to stop the action of NaOCl on dentinal structures which may weaken the root dentine (24, 25). These parameters were considered for the selection of materials in our study.

In the present study, the files were exposed dynamically to the selected irrigants with the manufacturer recommended settings to better simulate clinical conditions. Though NaOCl is shown to have significant advantage as a cleansing agent in endodontics, literature shows that it has some adverse effects such as corrosive actions including pitting or crevice corrosion on interaction with NiTi files which could potentiate to fracture of instrument in the long term (7). Both XP-endo Shaper and TruNatomy files showed increased surface values of Ra and RMS post exposure to different endodontic irrigants, Drastic increase of surface roughness was observed when both files were exposed to 17% EDTA in comparison to 5.25% NaOCl. These deleterious effects were found to be greatly reduced when exposed to 5.25% NaOCl and 17% EDTA with a resultant decrease in Ra and RMS values. This variation in results could be due to selected instruments undergoing different manufacturing processes which may have inherent characteristics to resist corrosion on exposure to irrigants. In contrast to the achieved results, the amount of surface roughness varied with different instruments (26). For instance, Topuz et al. (27) showed that RaCe files on exposure to NaOCl showed significant surface deterioration whereas Cai et al. (17) showed Hy-Flex files did not get affected by 5.25% NaOCl. This was not limited to NaOCl but also on exposure to 17% EDTA, Fayyad et al. (28) found that RaCe files and GTX files did not show much

surface changes on exposure to 17% EDTA but Prasad et al. (18) reported that iRaCe and ProTaper instruments showed marked surface changes on exposure to NaOCl and EDTA.

XP-endo Shaper is a NiTi file made with a proprietary 'MaxWire' technology. It has shown to combine both shape memory and superelasticity properties in clinical application (29). XP-endo Shaper has shown to exhibit "A" phase to "M" phase at room temperature with a variable taper. Recent studies (30, 31) have shown an increased levels of cyclic and torsional fatigue resistance compared to other file systems at intracanal temperature. Another recently introduced NiTi system, TruNatomy are heat treated files with a parallelogram shaped cross section showing an increased resistance to fatigue (15, 16). Though both files have proved to show increased fatigue resistance, its interaction against different irrigants is not proven till date. In the current study, overall surface roughness was found to be significantly lesser for XP-endo Shaper in contrast to TruNatomy files post-exposure to different irrigants. These results could be due to the files showing a varied NiTi structural composition and manufacturing process with the results seen contradictory to the previous studies.

Our study showed significantly higher surface morphology changes with TruNatomy files in comparison XP-endo Shaper when exposed to 5.25% NaOCl ($P < 0.05$), 17% EDTA ($P < 0.05$), 5.25% NaOCl+17% EDTA ($P < 0.05$). This could be due the influence of different manufacturing process which could exhibit various surface defects on the flutes of the files which is often tackled by manufacturers using surface treatment. Different manufacturers use different surface treatment techniques during the production process of endodontic files such as ion implantation, thermal nitridation, cryogenic treatment, nitride coating and electropolishing (32). XP-endo Shaper files are treated with electropolishing and could have potentiated to lesser surface roughness post-exposure to irrigants based on

the results observed in our study. Electropolishing alters the surface topographic characteristics of the file by inducing a uniform oxide layer over the surface making it resistant to corrosion or fracture by fatigue (33). In spite of its beneficial effect on the lifespan of the files, some authors have contradicted this and found that electropolishing has no influence on corrosion and fatigue of the files (34, 35). TruNatomy is a heat-treated alloy with no data available from the manufacturer about the surface treatment done on these files.

The present study has examined the files in in-vitro conditions which is considered as one limitation. A number of factors such as the amount of root dentine removed, canal curvatures may have significant effects on the overall fatigue levels of the file.

CONCLUSION

Within the limitations of the present study, it can be concluded that the overall surface roughness upon irrigant exposure was lesser in XP-endo Shaper compared to TruNatomy files. Exposure to 17% EDTA caused significantly higher surface roughness to both the files compared to 5.25% NaOCl. In contrast, both the tested files on exposure to root canal irrigants (5.25% NaOCl and 17% EDTA) in combination showed lesser roughness in comparison to exposure to individual irrigants. XP-endo Shaper exhibited overall lesser loss of elements (Ni and Ti) on exposure to different irrigants in comparison to TruNatomy files. Clinicians should use EDTA with caution with the examined file systems since it may cause deleterious effects on the surface causing crack propagation and subsequent instrument separation.

Disclosures

Conflict of interest: The authors deny any conflict of interest related and do not endorse or promote any of the companies of the files used in this study.

Ethics Committee Approval: The study was conducted after the approval of the ethical committee of Saveetha University with the approval number of SRB/SDC/ENDO-1805/20/01 dated 21/07/2020.

Peer-review: Externally peer-reviewed.

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