scientific reports

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Effect of phytic acid on chemical, structural, and mechanical characteristics of nickel–titanium endodontic files

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This study investigated phytic acid (IP6) effect on chemical, structural, and mechanical characteristics of nickel–titanium (NiTi) files. The tested files were equally divided into groups according to the immersion protocol: sodium hypochlorite (NaOCl), ethylenediaminetetraacetic acid (EDTA), IP6, EDTA followed by NaOCl, and IP6 followed by NaOCl. These groups were then compared in terms of Ni, Ti, and chromium (Cr) ions release from the files. Microstructural changes using field emission scanning electron microscope (Fe-SEM) and energy dispersive X-ray spectroscopy (EDX) and surface roughness were analyzed. The mechanical characterization was conducted using cyclic fatigue resistance test. Fractured segments were scanned under SEM. Statistical analysis was performed using one-way ANOVA, Tukey test, Kruskal–Wallis test and Mann–Whitney U test. Results showed that NaOCl caused significant release of Cr, followed by IP6 and EDTA ($P<0.05$). When files were pre-immersed in EDTA, NaOCl tended to induce less release of Ti and Cr. EDX evaluation revealed that the main surface elements were Ni, Ti, carbon, and oxygen. EDTA group contained the highest amount of carbon, while the control group showed the lowest. Surface roughness evaluation revealed no significant differences between groups despite the minor increases after immersion in certain groups. Black areas were observed in the NaOCl group which indicated corrosion. However, the cyclic fatigue test showed no significant differences between the groups.

Keywords Cyclic fatigue, EDTA, Ion release, Phytic acid, Sodium hypochlorite

The goal of root canal treatment is the elimination of bacteria from the root canal system and this is mainly achieved by mechanical instrumentation and chemical disinfection[1]. The former is accomplished with the use of files, and nickel–titanium (NiTi) endodontic files have gained popularity over the conventionally used stainless steel files due to the superior properties of the NiTi alloy. The super elasticity, flexibility, shape memory, and high torsional strength were amongst the reasons behind its quick adoption clinically and improved treatment outcomes[2].

Chemical agents are used to disinfect the root canals and sodium hypochlorite (NaOCl) is the most commonly used endodontic irrigant for this purpose[3]. NaOCl is known for its excellent soft tissue dissolving ability and potent antimicrobial ability against an array of endodontic microbes[4–6]. Despite these desired properties, it is not without shortcomings; NaOCl is a powerful oxidizing agent with high alkalinity that has the ability to corrode metals and thus may alter the mechanical properties of endodontics files[7].

Ethylenediaminetetraacetic acid (EDTA) is another popular agent used during root canal treatment to remove the smear layer and facilitate the mechanical action of files[8]. To achieve complete disinfection of the root canal system, a combination of irrigation and mechanical instrumentation is necessary. This entails NiTi files to be in contact with different irrigation solutions for the whole period of instrumentation[9], which raises concerns on the potential of corrosion of NiTi files. Moreover, files may be exposed to NaOCl during chairside disinfection of unused or contaminated instruments. Despite recommendations to limit NaOCl exposure to only 5 min, extended exposure, such as overnight soaking, may still occur in clinical practice[10,11].

Corrosion is the loss of mass of metallic materials over time as a result of interactions with the surrounding environment. It is a spontaneous electrochemical process that involves reactions of oxidation and reduction

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and occurs in corrosive environments that include acids, alkalis, and saline solutions. Corrosion can also occur when metals react with oxygen (O_2), hydrogen, bacteria, or when a metal is subjected to an electrical current[12]. The NiTi files are in constant contact with irrigants during the canal instrumentation. It was revealed that even short time contact can induce changes on the file surface represented as surface corrosion, micro-pitting and eventually crack formation[7,13]. NiTi file corrosion is affected by many factors such as chemical composition, microstructural homogeneity, and the presence of impurities on the file surface[14]. The NiTi file corrosion has been previously investigated using electrochemical analysis[15,16], elemental analysis of metal ions released from the files[17,18], scanning electron microscopy (SEM) analysis[19,20] and surface roughness[21,22]. It has been shown that corrosion influences the file performance inside the canal[7,16,19,23]. Therefore, understanding alloy corrosion is crucial for reducing the incidence of file fracture[24]. However, no research has yet examined the protective effects of potential anticorrosive agents on the surface characteristics and mechanical performance of NiTi files.

Chelating agents have received a lot of attention over the last few decades as inhibitors of corrosion[25]. Phytic acid, inositol hexakisphosphate, (IP6) is a naturally occurring substance that is present in plants and seeds[26]. IP6 offers a range of desired properties due to its unique structure and the highly negatively charged phosphate groups. It has been proposed as an anticorrosive agent due to its powerful chelating capability and the ability to coat or adsorb onto metal surfaces. The efficiency of IP6 as an anticorrosive agent is dependent on a number of parameters, including, but not limited to, the specific metal involved, the concentration of IP6, and ambient circumstances such as pH[27,28]. Thus, it is regarded as a promising alternative agent for the pre-treatment of alloys used in engineering[27,29]. However, research in this area is still ongoing and researchers are still investigating potential applications of this agent as corrosion inhibitor and further development are needed to fully understand and optimize the anticorrosive properties of IP6 for practical applications[27].

In the dental field, research on IP6 started several decades ago after the discovery of its cariostatic and enamel protection properties[30]. Despite the encouraging data, there was a period of diminished interest, which delayed research progress. However, the potential use of IP6 has lately been re-visited and new applications of IP6 have been investigated with findings that showed significant evidence of its benefits in dentistry[31,32]. In endodontics, IP6 research is still in its infancy but compelling data have been observed. IP6's readily availability, ease of extraction, biocompatibility, chelating activity and antimicrobial properties make it a promising alternative to EDTA as a root canal chelating agent[33,34]. Thus, the aim of this proof-of-concept research was to investigate for the first time the effect of IP6 on the chemical, structural, and mechanical characteristics of NiTi files and compare it to conventional agents, namely NaOCl and EDTA.

Materials and methods

Selection and preparation of the samples

A total of 126 brand new ZenFlex files (Kerr Corporation, Pomona, CA, USA) with a tip size 25, 0.06 taper, and length 25 mm were obtained. Forty-two files were used for elemental analysis of metal release from the files and microstructural characteristics analysis including surface roughness after immersing in different solutions. Eighty-four files were used for cyclic fatigue resistance. All files were inspected under ×8 magnification (A-6 Surgical Operating microscope, Global Surgical Cooperation, USA) for the presence of manufacturing defects. None of the files were discarded.

Elemental analysis of metal release from the files after immersing in different solutions

The sample size calculation was performed using an alpha of 0.05 and a power of 0.80. Based on these calculations, the sample size was determined as 42 files distributed in seven groups. Files were randomly and equally allocated according to Table 1, where distilled water group was considered as the control group as per the requirement for the method of analyzing metal ions release while files without immersion were used as the control for field-emission scanning electron microscope (Fe-SEM) (Apreo 2 SEM, Thermofisher Scientific, Massachusetts, USA) and energy dispersive X-ray spectroscopy (EDX) (Ultim Max EDS detector, Oxford Instruments, UK) examinations. Plastic tubes were used to immerse the files in 1.8 ml of each solution. A circular hole was drilled in the cap of each tube allowing the files to be inserted through the hole and immersed in the solution. Only the working part of the file (16 mm length from the tip) was allowed to immerse in the solution, while the shank of the file was not in contact with the solution to avoid galvanic corrosion as described in previous studies[7,10]. After the immersion period was reached, files were kept for microstructural analysis, and the solutions in which the files were immersed were collected for elemental analysis for the presence of metal ions released from the files.

Group	Type of solution and immersion time
Control	1. Distilled water (Thermo Fisher Scientific, USA) for 1 h for metal release (n = 6) and 2. un-immersed files for FE-SEM and EDX (n = 6)
NaOCl	5.25% NaOCl (pH 12.62) (Cerkamed Medical Company, Stalowa Wola, Poland) for 1 h
EDTA	17% EDTA (pH 7.23) (Cerkamed Medical Company, Stalowa Wola, Poland) for 10 min
IP6	2.5% IP6 (pH 1.00) (Sigma-Aldrich, Massachusetts, United States) for 10 min
EDTA/NaOCl	17% EDTA for 10 min, followed by 5.25%NaOCl for 1 h
IP6/NaOCl	2.5% IP6 for 10 min, followed by NaOCl for 1 h

Table 1. Experimental and control groups (n = 6).

Elemental analysis was done using spectrometer (ICP Spectrometer, iCAP 7000 series, Thermofisher Scientific, USA). The metals tested in the samples were Ni, Ti, and chromium (Cr). To quantify the amounts of metal ions released from the files into the solution, the amount of Ni, Ti and Cr was determined in the solution before and after immersion of the files. The following equation was used to determine the amount of metals released in each solution:

$$\text{Metal}_{\text{released}} = \text{Metal}_{\text{after immersion}} - \text{Metal}_{\text{before immersion}}$$

Evaluation of microstructural characteristics, elemental analysis, and surface roughness of NiTi files after immersion in different solutions

After elemental analysis of metal release from the files, the files were rinsed with distilled water and dried followed by scanning under Fe-SEM for microstructural characteristics analysis and surface roughness, and new un-immersed files were used as the control group (as per Table 1).

The files were fixed on a metal plate, the 16 mm length of each file was divided into three parts and scanned under the following magnifications: coronal third (×100), middle third (×150), apical third (×300 and ×1000). Elemental analysis of the file surface was carried out using EDX. The acceleration voltage was set to 10.00 kV for all the files. One reading from each third of the working part of the file was recorded.

Surface roughness was conducted following the methodology of Almohareb et al.[35] at ×1000 magnification. The images were taken in the apical third of the file (i.e. the third flute from the tip) using Fe-SEM and then analyzed using ImageJ 1.53t software (NIH, Bethesda, MD, USA) to evaluate the topographic changes of the files. The values of the arithmetic average roughness (Ra) were recorded from three locations per scan and the average was calculated.

Cyclic fatigue testing

Sample size calculation was conducted based on Alcalde et al. study[36]. The alpha-type error of 0.05, power of 0.95, and N2/N1 ratio of 1 were assumed. This showed a minimum sample size of eight files for each group. Therefore, 84 new ZenFlex files 25/0.06, 25 mm were divided equally, and randomly allocated into 6 groups (n = 14) similar to those in Table 1 with a control group of new un-immersed files. The files were immersed in solutions using the same method employed in the elemental analysis described above. After immersing the files, they were rinsed with distilled water and allowed to air dry before testing. The methodology used to test cyclic fatigue resistance was previously described by Jamleh et al.[37]. Briefly, a metallic simulated canal was used with curvature angle and radius of 60 degrees 5 mm, respectively. The maximum curvature was located 5 mm from the file end. An endodontic motor handpiece (X-Smart Plus; Dentsply Sirona) was set up in a stable position that allowed reproducibility for all files wherein each file was inserted inside the canal until 19 mm from its end and rotated at a speed of 500 rpm and a torque of 3 N cm until fracture was detected visually or audibly. The metallic canal was submerged inside the water bath with a controlled temperature of 37 °C. The number of cycles to fracture (NCF) was calculated by dividing the time to fracture (sec) by 60 and multiplying it by 500. The fracture surfaces of two files from each group were investigated using SEM at ×300 to evaluate surface characteristics.

Statistical analysis

Shapiro–Wilk test was used to test the data normality. One-way ANOVA, Tukey test, Kruskal–Wallis test and Mann–Whitney U test were run to analyze the data based on their normality using Shapiro–Wilk test. SPSS 22.0 (IBM-SPSS Inc., Armonk, NY, USA) was used at 5% significance level.

Results

Elemental analysis of immersing solutions

A summary of the levels of metals in parts per billion (ppb) detected in the solutions are reported in Table 2. Spectrometry analysis of the solutions in which files were immersed revealed quantifiable traces of Ni in the EDTA, IP6, and distilled water (control) groups only. The IP6 group contained significantly higher Ni levels when compared to the EDTA group ($P < 0.05$). While EDTA and distilled water groups were not significantly different from each other ($P > 0.05$). Spectrometry showed Cr to be present in all groups except the distilled water group. The highest amount of Cr was detected in the NaOCl group, which was significantly higher than EDTA and IP6 groups ($P < 0.05$). NaOCl group also showed higher amounts of Cr compared to EDTA/NaOCl and IP6/NaOCl; however, this did not reach a level of statistical significance ($P > 0.05$). The latter two groups also showed comparable Cr levels ($P > 0.05$). Ti ions were detected in NaOCl, IP6, EDTA/NaOCl, and IP6/NaOCl, but not in EDTA group. The IP6 group showed the highest amounts of Ti, while the EDTA/NaOCl group showed the lowest.

Type of element	NaOCl	EDTA	IP6	EDTA/NaOCl	IP6/NaOCl	Control (distilled water)
Nickel	ND	0.60 (0.53)[a]	2.53 (1.93)[b]	ND	ND	1.14 (0.80)[ab]
Chromium	1.71 (0.77)[c]	0.14 (0.33)[a]	0.65 (0.44)[ab]	1.25 (0.59)[bc]	0.95 (0.52)[abc]	ND
Titanium	1.98 (1.40)[ab]	ND	4.11 (0.81)[b]	0.59 (0.84)[a]	2.07 (2.23)[ab]	ND

Table 2. Mean (SD) of nickel, chromium, and titanium (in ppb) in the tested groups. Within a row, different letters indicate statistically significant difference ($P < 0.05$). ND: not detected.

The IP6/NaOCl group showed comparable Ti levels to the NaOCl, IP6 and EDTA/NaOCl groups ($P > 0.05$). The EDTA/NaOCl group showed a low Ti level that was statistically insignificant compared to the NaOCl.

Evaluation of microstructural characteristics, surface roughness, and elemental analysis of NiTi files after immersion

Fe-SEM and EDX analysis

Representative Fe-SEM images from each group are shown in Figs. 1, 2, and 3 for the apical, middle and coronal thirds of the files; respectively. Fe-SEM images of the control group (new un-immersed files) showed few surface irregularities and defects. Metal rollover was noticed along the cutting blades of some of the files. Compared to the control group, the NaOCl group showed areas of black residues on the files surfaces, particularly at the apical portion of the files. These residues are suggestive of corrosion and corrosive by-products accumulating on the file

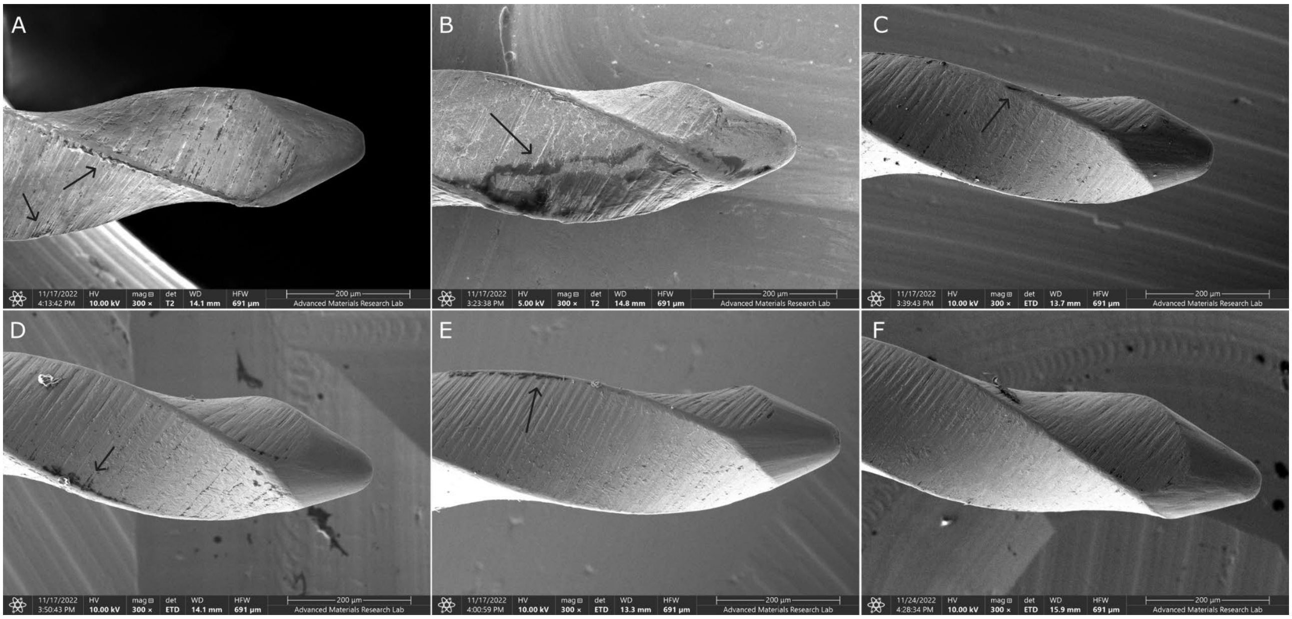

Figure 1. (**A**–**F**): Representative Fe-SEM images of groups showing scans from the apical portion of the working part of the file. Arrows point to surface defects and corrosive changes on the file surface. (Magnification ×300) (**A**: Control, **B**: NaOCl, **C**: EDTA, **D**: IP6, **E**: EDTA/NaOCl, **F**: IP6/NaOCl).

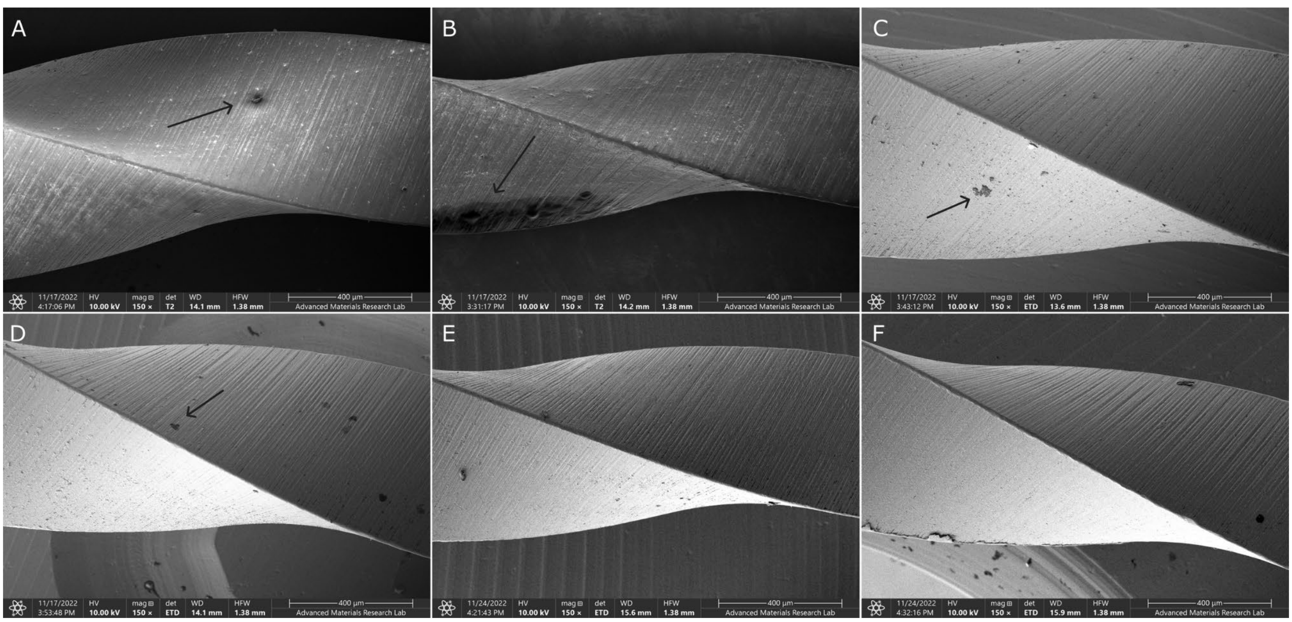

Figure 2. (**A**–**F**): Representative Fe-SEM images of groups showing scans from the middle portion of the working part of the file. Arrows point to surface defects and corrosive changes on the file surface. (Magnification ×300) (**A**: Control, **B**: NaOCl, **C**: EDTA, **D**: IP6, **E**: EDTA/NaOCl, **F**: IP6/NaOCl).

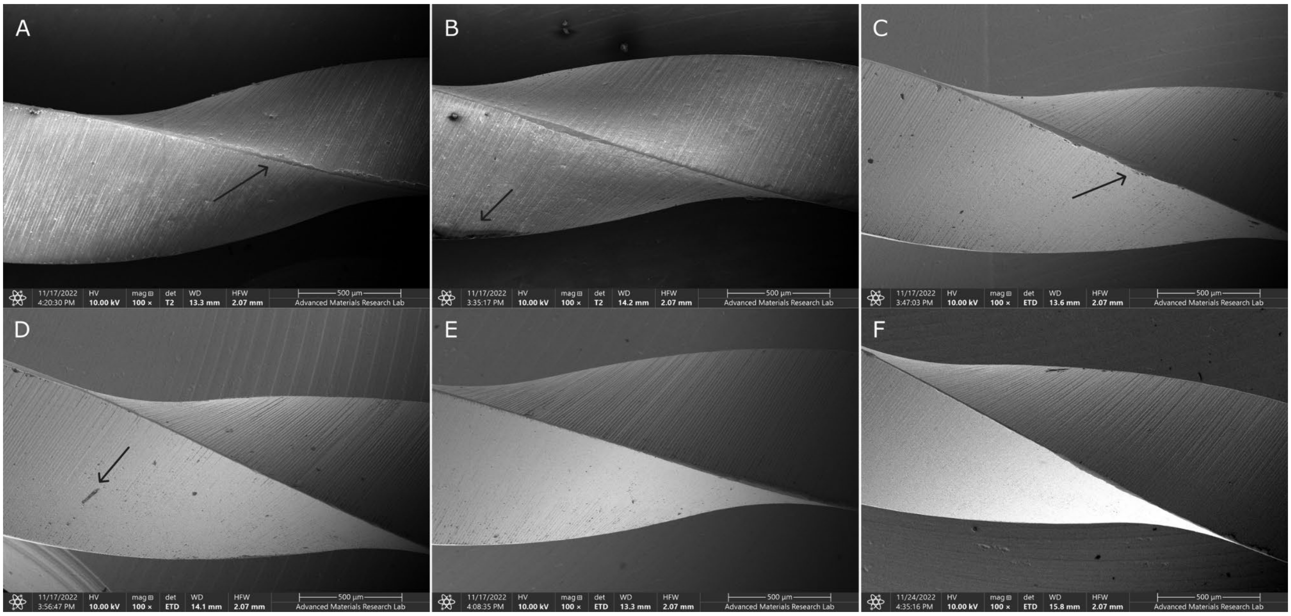

Figure 3. (**A–F**): Representative Fe-SEM images of groups showing scans from the coronal portion of the working part of the file. Arrows point to surface defects and corrosive changes on the file surface. (Magnification ×300) (**A**: Control, **B**: NaOCl, **C**: EDTA, **D**: IP6, **E**: EDTA/NaOCl, **F**: IP6/NaOCl).

surface. Corrosion was seen to be more present along the cutting blade of the file. Fe-SEM images of EDTA and IP6 groups showed fewer black residues compared to NaOCl group. In the combined exposure groups (EDTA/NaOCl or IP6/NaOCl) less signs of corrosion were detected in comparison to the NaOCl group. Similar to the findings of the NaOCl group, corrosion was seen more in the apical portion compared to the middle and coronal thirds in the other experimental groups.

The main elements present on the file surface were Ni, Ti, carbon (C), and O_2. The mean atomic weight percentages for the elements in all groups are summarized in Table 3. Cr was identified on the surfaces of files in all groups but not in all samples with a maximum weight percentage of 11.5%. Other elements were scarcely detected in the control group (new un-immersed files) included calcium, and nitrogen. While the NaOCl group contained other elements like iron, and magnesium (Mg). Both EDTA and IP6 groups contained small amounts of Mg, aluminum (Al), and phosphorus. EDX microanalysis of the EDTA/NaOCl group also showed Mg and Al. Whereas analysis of files from the IP6/NaOCl group did not reveal any metals other than Ni, Ti, C, and O_2. Silicon was consistently detected on most of the files from all groups.

Statistical analysis showed that there was no significant difference in the amount of Ni and Ti on the file surface between any of the groups. When the percentages of C were compared between groups, NaOCl and EDTA groups showed the highest mean levels of C. However, only EDTA group contained significantly higher C levels compared to the control group ($P < 0.05$). The EDTA group also showed significantly higher C levels compared to both EDTA/NaOCl and IP6/NaOCl ($P < 0.05$). There was no difference in the O_2 levels between any of the groups ($P > 0.05$).

Surface roughness of files

The mean and standard deviation of the surface roughness values are shown in Fig. 4. The average surface roughness of the control group (new un-immersed files) was 14.29 ± 1.75 um. The changes in surface roughness were minimal and did not reach a level of statistical significance between any of the groups. When comparing the

Group	Nickel	Titanium	Carbon	Oxygen
Control	54.57 (3.08)[a]	40.55 (4.12)[a]	2.88 (1.46)[a]	4.36 (0.86)[a]
NaOCl	53.69 (4.32)[a]	35.72 (3.44)[a]	4.67 (1.16)[ab]	4.08 (0.98)[a]
EDTA	52.58 (3.98)[a]	35.58 (5.14)[a]	5.18 (2.63)[b]	4.72 (0.99)[a]
IP6	54.73 (2.50)[a]	38.2 (3.75)[a]	4.03 (1.48)[ab]	3.72 (0.73)[a]
EDTA/NaOCl	55.58 (2.50)[a]	35.73 (3.67)[a]	4.03 (1.28)[a]	4.53 (1.12)[a]
IP6/NaOCl	55.08 (3.03)[a]	38.73 (4.45)[a]	3.18 (0.98)[a]	4.43 (0.76)[a]

Table 3. Mean (SD) of the elemental analysis of files after immersion in different irrigation solutions. Within a column, different letters indicate statistically significant difference ($P < 0.05$).

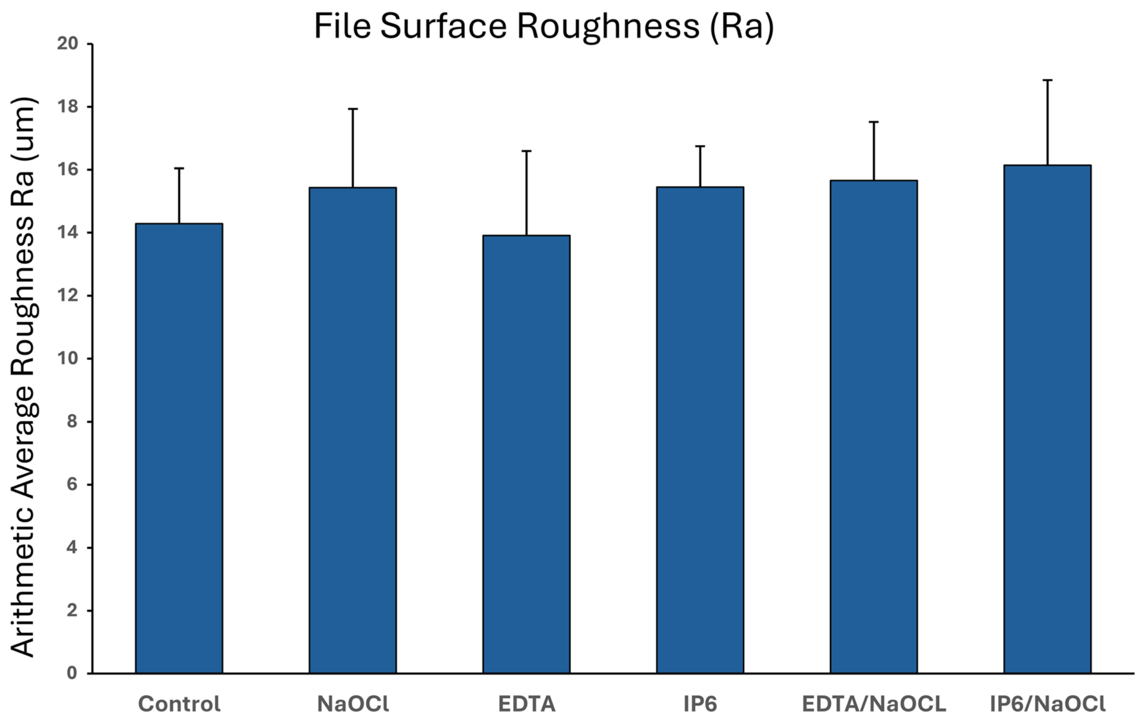

Figure 4. Mean (±SD) of surface roughness of files in different immersion protocols.

different solutions, EDTA was the only solution that resulted in lower surface roughness compared to the control group despite the insignificant statistical difference.

Cyclic fatigue test

The mean and standard deviation of NCF and fractured segment length are presented in Table 4. Considering the NCF, no significant difference was noted ($P > 0.05$). When comparing the length of the fractured segment, EDTA group exhibited the shortest fragment, while the longest was observed in the EDTA/NaOCl group; however, no significant differences were detected among the groups ($P > 0.05$). SEM evaluation revealed typical signs of cyclic fatigue in the fractured files where crack initiation area was evident at the edge, and fatigue striations along with dimpling were noted on the fracture surface. In the control samples, an area of brittle fracture was observed at the edge where the crack was initiated. All of these are typical signs of fatigue failure (Fig. 5), leading to the observation that all files fractured due to cyclic fatigue. It is worthy to note that the files in the NaOCl, EDTA, and IP6 groups had a smaller crack initiation area but larger crack propagation area when compared to the control group. While the files in the EDTA/NaOCl and IP6/NaOCl had a larger crack initiation area but smaller crack propagation when compared to the control group.

Selected samples were randomly chosen for EDX analysis of the fractured segments, it is noteworthy to note that the C content on the outer surface was higher than the core of the file, while, the opposite was observed for the Ni and Ti levels (Table 5). Meanwhile, Cr was present in the core and outer surface of some files with quite similar values; however, not all samples showed Cr in the EDX analysis (data not shown).

Discussion

The corrosion of NiTi files has long been a research focus, particularly given the aggressive nature of certain irrigation solutions used in endodontics. Despite the extensive studies on corrosion, there has been no effort to explore the potential of anti-corrosive agents to counteract this issue. The aim of this proof-of-concept study

Group	Number of cycles to fracture	Fractured segment length (mm)
Control	217.26 (51.0)	5.61 (0.46)
NaOCl	292.26 (64.67)	5.73 (0.35)
EDTA	252.38 (70.82)	5.47 (0.52)
IP6	272.62 (86.63)	5.50 (0.50)
EDTA/NaOCl	247.02 (76.00)	5.78 (0.44)
IP6/NaOCl	252.39 (91.78)	5.66 (0.46)

Table 4. Mean (±SD) of cyclic fatigue data for files subjected to different immersion protocols.

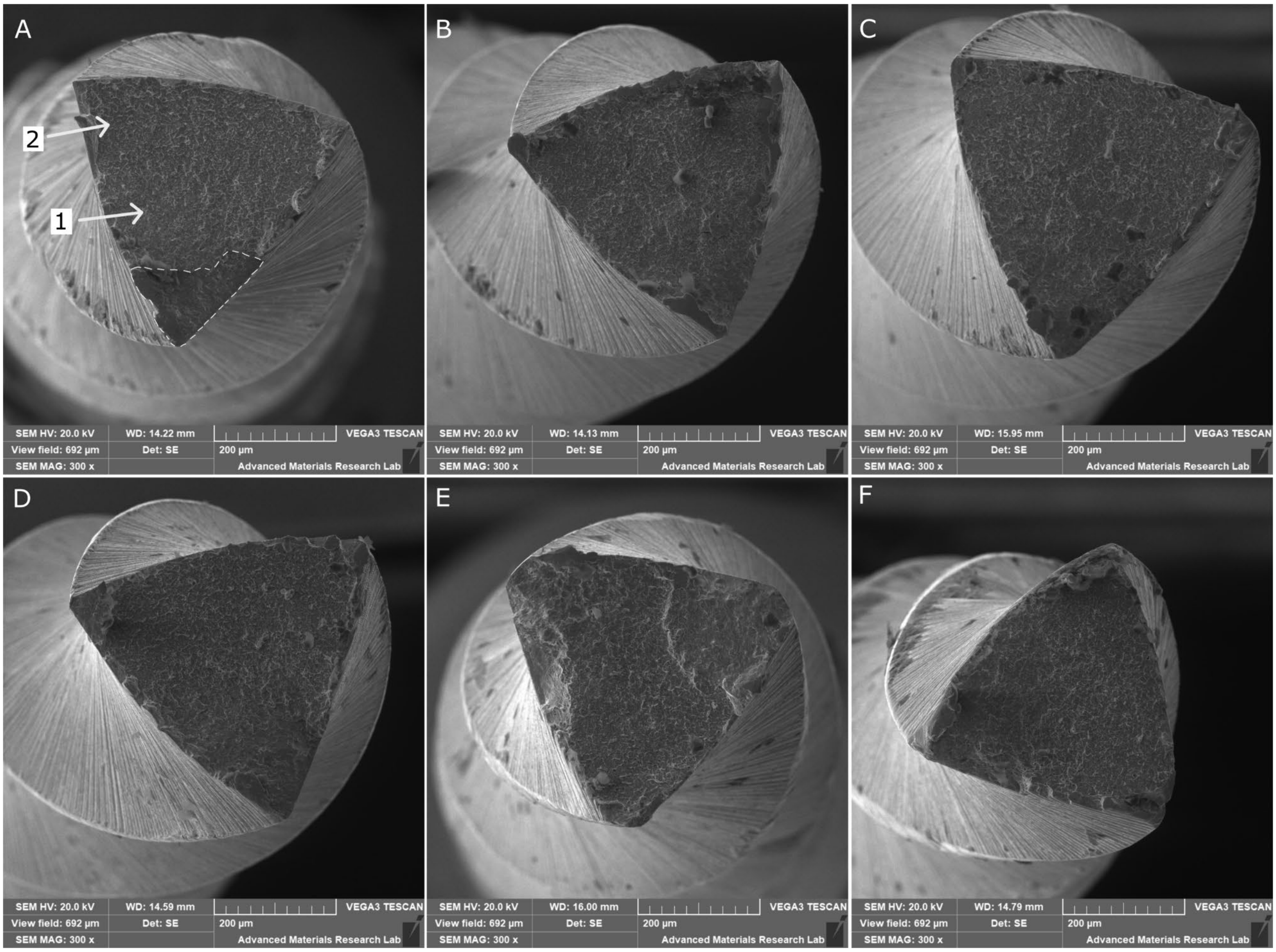

Figure 5. (**A**–**F**): SEM images of files after cyclic fatigue testing in different solutions. **A**: Control, **B**: NaOCl, **C**: EDTA, **D**: IP6, **E**: EDTA/NaOCl, **F**: IP6/NaOCl. Magnification ×300 was used for all samples. Dotted area represents crack initiation site. Area 1 represents crack propagation (characterized by elongated striations), while area 2 shows the site of ductile failure and file separation.

	Core			Outer surface		
	Nickel	Titanium	Carbon	Nickel	Titanium	Carbon
Control	51.2	43.15	5.65	37.4	29.25	18.55
NaOCl	48.55	41.35	10.1	42.1	31.4	15.55
EDTA	50.5	40.7	8.8	46.75	35.1	18.1
IP6	52.75	40.45	6.85	45.35	36.5	18.15
EDTA/NaOCl	51.25	40.25	8.5	39.25	30	21.95
IP6/NaOCl	52.1	42.3	5.6	36.8	27.5	23.55

Table 5. EDX elemental percentages for the core and outer surface of the files.

was to investigate the effect of immersing NiTi files in IP6 on their mechanical, structural, and chemical characteristics. We also compared the findings with those of commonly used irrigants namely NaOCl and EDTA.

Previous corrosion studies have typically focused on a single aspect of corrosion evaluation. In contrast, the present study conducted multiple investigations to assess the corrosion process encompassing chemical, structural, and mechanical changes. The first investigation included quantifying the amounts of metals released from endodontic files during the immersion in different solutions. This concept has been applied to study corrosion of metal alloys in the fields of implant dentistry and engineering[38,39].

Under the conditions of the present study, it has been noticed that corrosion did occur in all solutions tested, as different metals were dissolved from the tested files in different quantities depending on the used solution and the immersion protocol. Consistently, it was recently reported that NiTi file exhibited greater corrosion rates in NaOCl[24]. Moreover, when the files were pre-immersed in EDTA or IP6, NaOCl showed a tendency to cause less release of certain metal ions. This supports the hypothesis that chelating agents can form stable complexes with metals which might prevent further corrosion in the subsequent immersion in NaOCl[16,40–42]. This can also be attributed to the stability of the re-passivation layer formed during the process of de-passivation and re-passivation of metals[42]. This warrants further investigation on the probable role of EDTA and IP6 in minimizing the deleterious effect of NaOCl on the file surface. The cut off level of metal ions release that would affect the integrity of the surface and the mechanical performance of the files has not been reported in the literature and is an area of research to be explored in the field of endodontics.

Quantitative data on the corroded metals from NiTi files is scarce in the literature, as most studies focused on investigating visual signs of corrosion using SEM and atomic force microscopy, and assessing the impact of corrosion on mechanical performance through fatigue resistance testing. Corrosion of Lightspeed files when immersed in 1% or 5% NaOCl was investigated in 1998. Similar to the findings of our study, immersing the files in 5% NaOCl for 1 h resulted in the release of Ti in the NaOCl solution[17]. Topuz et al. demonstrated surface pitting and cracks on NiTi files when immersed in 5.25% NaOCl for 5 min[43]. Toker et al. reported that quantifiable amounts of Ni ions were released when various types NiTi files were immersed in simulated body fluid. They observed higher Ni release in files with greater surface defects[18], and this validates previous findings suggesting that surface irregularities act as high-energy sites that predispose the files to corrosion and elevated ion release[44].

Our results indicated the release of Cr from the files alongside Ni and Ti. Manufacturers typically do not list Cr as part of the composition of NiTi alloy used to produce endodontic instruments. However, Cr is known to constitute 17–20% of the composition of manual stainless-steel files[45]. In metallurgy, the addition of Cr to NiTi alloy has been reported to increase rigidity and hardness and reduce transformation temperature[46]; this could potentially account for the presence of Cr in the metal dissolution and EDX analyses of the current investigation.

To further investigate the clinical significance of the corrosion observed in our study, the immersed files were examined under Fe-SEM to observe surface microstructural changes. Our examination revealed that new Zenflex files displayed minimal manufacturing defects, with the occurrence of these flaws being more pronounced at the apical third of the file. Overall, most files showed manufacturing defects and machining marks, this represents an area for improvement in the current machining processes and file manufacturing methods[47,48]. Our scans revealed the presence of black residue of corrosion by-products precipitating on the file surface. These residues were most prominent in the NaOCl group, less so in the EDTA and IP6 groups, and least evident in the EDTA/NaOCl and the IP6/NaOCl groups. Our findings on the black precipitate were consistent with the findings of Han-Hsing et al. where SEM observations after immersing BioRace, TRUshape, and EdgeFile in 4% NaOCl for 24 h revealed black precipitates on the files surfaces[20]. Previous studies reported visual signs of corrosion on SEM examination using various concentrations, temperatures, and immersion durations of NaOCl and EDTA, as well as different file systems[9,49,50]. Whereas many other studies did not detect evidence of corrosion on SEM examination of files subjected to irrigation solutions[16,51,52]. Factors influencing the corrosion resistance of the files include many variables, including file surface treatment, geometry, cross section, and design[20]. Alloy composition such as Ni and Ti levels, alloy impurities, microstructural homogeneity, and surface residue also play salient role[10,14,53].

In our EDX analysis, all experimental groups showed higher C levels on the files surface compared to the control group, with EDTA only being statistically significant. While most metal studies typically consider the presence of C as a form of contamination[54,55], there is a distinct perspective regarding its significance in NiTi orthodontic wires where it has been postulated that an increase in C content in these wires may signify the formation of titanium carbide[56]. An investigation on the corrosion and passivity behaviors of NiTi shape memory alloy used in biomedicine revealed a correlation between lower C levels and enhanced corrosion resistance. This association stems from the fact that C content influences the formation and dispersion of titanium carbide particles. Oxides generated at this interface are deemed relatively unstable and non-protective, rendering the metal susceptible to pitting[57]. Our EDX analysis indicated that the control group exhibited the lowest C weight percentages, followed by the IP6/NaOCl group.

The third part of this investigation included analyzing alterations in files surface roughness. It has been previously established that surface topography of NiTi files influences the fracture mechanism[58], as surface irregularities serve as nucleation points for crack formation and propagation leading to failure[59]. Our results indicated that changes in surface roughness following immersion of files in different solutions were negligible and did not reach a level of statistical significance. Two previous studies reported a decrease in Ra values of WaveOne Gold and Reciproc Blue files after root canal preparation in simulated canals irrigated with 3% NaOCl. Although this decrease in roughness was not statistically significant; the authors attributed this to the polishing effect resulting from the file contact with the artificial canal[60,61]. Other studies, on the other hand, reported that immersion in NaOCl and EDTA increases the surface roughness of the files as a result of corrosion[13,21]. Comparing results directly across studies investigating alterations in the surface topography of files poses challenges due to significant variations in the manufacturing process of the files, surface treatments, metallurgical compositions, pH levels of the tested solutions, and methodologies employed for evaluating surface roughness. The surface topography changes observed in this study cannot be completely interpreted within the scope of the current investigation, and hence this calls for further studies on the effect of each variable mentioned above on the corrosion resistance of NiTi files.

From a clinical standpoint, comprehending the impact of corrosion on file performance is crucial as it could potentially result in file fracture within the canal during preparation[24]. A recent study revealed certain effects of irrigants on the cutting efficiency of files, as indicated by corrosion, microcracks, and crevices observed on the tested files following disinfection with NaOCl[62]. The performance of NiTi files can be evaluated in terms of their resistance to cyclic fatigue. Despite subjecting the tested files to various immersion protocols aimed at simulating clinical conditions where files are continuously exposed to chemicals during canal instrumentation, no significant differences between the groups were observed in our study. Similarly, previous research has consistently reported no decrease in cyclic fatigue resistance following immersion in irrigants like NaOCl and EDTA[63–65]. Conversely, other studies have suggested that exposure of files to these agents may induce pitting, potentially compromising cyclic fatigue resistance and increasing the risk of file fracture[19,50,51,66,67]. These discrepancies might be partly attributed to the use of different file systems. Prior research has indicated a potential inclination towards greater corrosion resistance in heat-treated files when contrasted with their non-heat-treated counterparts, attributed to the formation of a passivation layer[9,24]. Furthermore, endodontic files are usually tested under low cycle fatigue where it rotates in severely curved canal until fracture. This testing setup may not accurately reflect the true impact of corrosion on file performance. Moreover, the absence of standardization in cyclic fatigue testing conditions, solution concentrations, and immersion durations renders the comparison of our results with previously published investigations impractical.

Conclusion

The limitations of this study include but are not limited to the use of in vitro experimental conditions which contribute to only limited answers to more complex problems. Moreover, this investigation included static immersion of files in irrigation solutions which might not reflect the dynamic nature of the clinical settings, and the interactions of the irrigation solutions with the dentin present in the canal which can lead to changes in the pH of the solutions. Within these limitations, we found that signs of corrosion occurred in all experimental solutions as per the metal release and Fe-SEM observation, but this did not reflect an effect on the cyclic fatigue resistance of the files and surface roughness. Exposure of the files to EDTA or IP6, before immersing in NaOCl showed a tendency to reduce the amount of certain metal ions released from the files by the action of this oxidizing agent. While these findings are of interest, their clinical relevance remains to be determined and further research is warranted to understand the effect of chelating agents on the corrosion resistance behavior of endodontic files.

Data availability

The authors confirm that the data supporting the findings of this study are available within the article and raw data are available on request from the corresponding author.

Received: 14 January 2024; Accepted: 9 August 2024
Published online: 29 August 2024

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Acknowledgements
We are thankful for the support of the Office of Vice Chancellor for Research and Graduate Studies, University of Sharjah, Sharjah, United Arab Emirates. We are also grateful for Professor Mohammad Harb Semreen, Dr Waad Kheder and Mr. Muath Mousa for their insights and help with the metal release experiment using the ICP spectrometer.

Author contributions
Conceptualization: M.N. and A.J. Methodology: M.S., M.N., A.A., H.A., and A.J. Software: M.N. and M.S. Validation: M.N. and A.J. Formal analysis: M.N., M.S., and A.J. Investigation: M.N. and M.S. Resources: M.N. and A.J. Data Curation: M.S., M.N., A.A., H.A., and A.J. Writing-Original Draft: M.S. and M.N. Writing-Review & Editing: M.S., M.N., A.A., H.A., and A.J. Visualization: M.S. and M.N. Supervision: M.N. and A.J. Project administration: M.N.

Funding
This study was funded by University of Sharjah, Research Project No. (2201100262). The funder had no role in study design, data collection and analysis, decision to publish, or preparation of the manuscript.

Competing interests
The authors declare no competing interests.

Additional information
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