RESEARCH ARTICLE

CYPRUS
JOURNAL OF MEDICAL SCIENCES

DOI: 10.4274/cjms.2021.2475
Cyprus J Med Sci 2023;8(2):136-141

The Determination of the Corrosion Rates of Rotary Ni-Ti Instruments in Various Irrigation Solutions

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Abstract

BACKGROUND/AIMS: This study's goal was to assess and make a comparison of the electrochemical corrosion rates of nickel-titanium (Ni-Ti) rotary files in cases of their immersion in four various irrigation solutions. Another goal of this research was to investigate and compare the areas of corrosion on the file surface under a scanning electron microscope (SEM).

MATERIALS AND METHODS: The Tafel extrapolation method was employed to perform the electrochemical determination of the corrosion rates of twenty-nine ProTaper Universal Ni-Ti rotary files in 2.5% sodium hypochlorite (NaOCl), 5% NaOCl, 15% ethylenediaminetetraacetic acid (EDTA) and 2% chlorhexidine gluconate (CHX) irrigation solutions (7 files were tested for each irrigant). Data were acquired by utilizing a combined system which contained a voltage scan generator, a potentiostat, and a recorder. In order to find corrosion rates, an extrapolation to corrosion potentials of the linear region of anodic currents acquired from electrochemical current-potential curves was performed. The Kruskal-Wallis One-Way analysis of variance was performed to analyze the data statistically. One randomly selected file from each test group was examined under SEM.

RESULTS: The corrosion rates of Ni-Ti rotary files in the examined solutions under SEM from the maximum to the minimum were as follows: 5% NaOCl >15% EDTA >2.5% NaOCl >2% CHX.

CONCLUSION: The findings of the present research demonstrated that 5% NaOCl, 15% EDTA, and 2.5% NaOCl led to significant corrosion on the surface of the chosen Ni-Ti rotary files.

Keywords: Chlorhexidine gluconate, corrosion, EDTA, Ni-Ti rotary files, sodium hypochlorite

INTRODUCTION

Endodontic treatment involves several instruments used with various irrigation solutions for chemo-mechanical preparation procedures.[1,2] Endodontic files produced from nickel-titanium (Ni-Ti) alloy are commonly utilized for endodontic instrumentation with various cross-sectional shapes, fabrication procedures, and design concepts, improved cutting efficiency, high torsional strength, and flexibility.[2,3] Despite numerous advantages, there is a risk of breakage in the course of root canal instrumentation regarding these files.[4] Ni-Ti endodontic rotary instruments usually fracture through either one or a combination of two mechanisms: torsional stress which is caused by the continuing rotation while the tip of the instrument binds in the canal, and flexural stress (bending stress) which arises by repeated compressive rotation of the instrument.[5] In addition to the repeated stresses on endodontic files, the corrosive environment within the

To cite this article: Özcan T, Sonat B, Dartar Öztan M, Basmaci F, Aksoy U. The Determination of the Corrosion Rates of Rotary Ni-Ti Instruments in Various Irrigation Solutions. Cyprus J Med Sci 2023;8(2):136-141

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Received: 21.07.2020
Accepted: 13.03.2021

root canal may cause instrumental fatigue during chemo-mechanical preparation.[2] Pitting or crevice corrosion affects metallic surfaces negatively and reduces the cutting efficiency of endodontic files, and thus promotes instrument breakage.[1] The corrosion mechanism of endodontic files during chemo-mechanical instrumentation stems from the disinfection and sterilization of the material, the pH, the temperature of the media, or the irrigation procedures.[5-8] Various endodontic irrigants at different concentrations, including NaOCl, citric acid, ethylenediaminetetraacetic acid (EDTA), and chlorhexidine gluconate (CHX), are used as disinfectant agents. Irrigants act as a lubricant during root canal instrumentation and they are necessary for the removal of debris created during usage. They also act as a solvent for tissues, as an agent for the promotion of root canal sterility, and act as a generator of open dentinal tubules on the root canal walls. Despite the numerous benefits of these solutions, the electrochemical and chemical aggressiveness of irrigants on endodontic instruments should also be considered in order to prevent a reduction of the lifespan of instruments with use.[9]

The hypothesis evaluated in this study was that corrosion rates, triggered by contact between metals and different electrochemical activities in the presence of NaOCl, EDTA or CHX may alter the structural integrity of the surface of a ProTaper Universal Ni-Ti file. The current research aimed to assess and make a comparison of the impact of 5% NaOCl, 2.5% NaOCl, 15% EDTA, and 2% CHX solutions on the electrochemical corrosion rates of ProTaper Universal Ni-Ti rotary files, by means of analysis via scanning electron microscope (SEM).

MATERIAL AND METHODS

The testing of twenty-nine new (unused) Ni-Ti rotary endodontic instruments (ProTaper Universal F3 ISO size 25 mm (6% taper), Dentsply Maillefer, Ballaigues, Switzerland) was performed for this research. All files were inspected under a stereomicroscope (S8 APO; Leica, Wetzlar, Germany) for defects and replaced if any defect was detected. In the control group, the instrument were not immersed in irrigation solution (n=1). The instruments were separated into four groups with seven specimens in each group. The irrigation solutions studied were as follows:

1. 2% CHX (pH 6.5) (Drogsan, İstanbul, Türkiye),

2. 5% NaOCl (pH 12.9) (Wizard, RehberKimya, İstanbul, Türkiye),

3. 2.5% NaOCl (pH 9.1) (Wizard, RehberKimya, İstanbul, Türkiye),

4. 15% EDTA (pH 7.5) (Wizard, RehberKimya, İstanbul, Türkiye).

The Tafel extrapolation method was employed for the electrochemical determination of the corrosion rates of the ProTaper Universal Ni-Ti rotary files placed in the irrigation solutions (Figure 1). Electrochemical tests were performed in a three-compartment Pyrex cell at a stable temperature of 37 °C. The cell was water-jacketed, and there was a connection with a constant temperature circulator. ProTaper Universal Ni-Ti rotary files were utilized as experimental electrodes. The files' cutting flutes were placed in the irrigation solutions and allowed to stand for a period of 20 min in the cell before every test in order for the electrode's rest potential to be achieved. A saturated calomel electrode (SCE) was utilized as a reference, while a platinum plate was utilized as a counter electrode. All potentials were referred to the SCE. In the course of the experiments, the continuous mixing of the solutions was carried out by a magnetic stirrer in order to allow for full contact of the solutions being tested with the entire surface of the files.

Data were acquired by utilizing a combined system which contained a voltage scan generator (Wenking VSG 72, Göttingen, Germany), a potentiostat (Wenking LB 75 L, Göttingen, Germany), and a recorder (Yokogawa 3077, Tokyo, Japan). The potential scan rate was selected to be 2.5 mV s^{-1}.

Statistical Analysis

To find the corrosion rates, the extrapolation (Tafel extrapolation) to corrosion potentials of the linear part of anodic currents, acquired from electrochemical current potential curves (E $\log_i$), was performed. Fresh solutions were used for every file. Statistical analysis was conducted with the Kruskal-Wallis One-Way ANOVA. The differences were considered statistically significant when $p<0.001$. One randomly selected file from each test group was kept for surface inspection in a special box to prevent its contact with any other medium at the end of the test. The said files, chosen in a random way, and an unused file (control sample) were examined under SEM and displayed at x250 and x500 magnifications.

RESULTS

Table 1 shows the corrosion rates of ProTaper Universal Ni-Ti instruments in 4 various irrigation solutions. The files' corrosion rates in all of the tested solutions were found to differ statistically significantly ($p<0.001$). The results indicated that the corrosion rates of ProTaper Universal Ni-Ti rotary instruments were at a maximum in the 5% NaOCl solution ($p<0.001$). Among the other solutions, the corrosion rates were revealed to be higher in the 15% EDTA than the 2.5% NaOCl and the 2% CHX ($p<0.001$) solutions. The lowest corrosion rate was determined in the 2% CHX solution.

The SEM microphotographs of the control and the four experimental groups were taken and investigated. In the SEM study on the unused control file, very few defects or debris were found due to the production stage of the file (Figure 2). It was seen that the Ni-Ti file which was

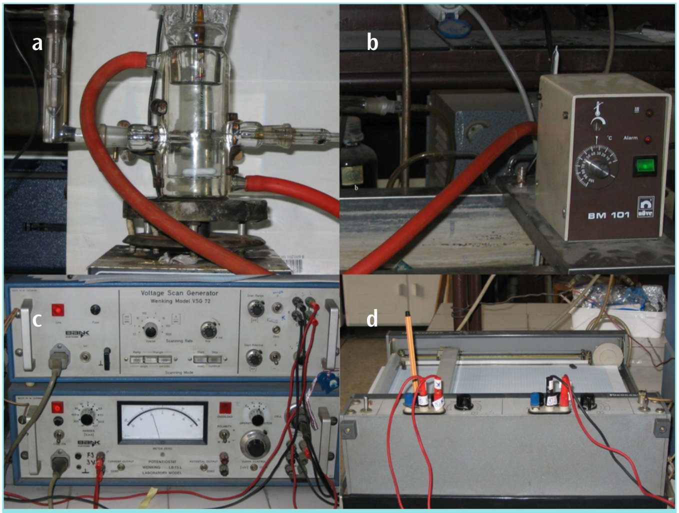

Figure 1. The Tafel extrapolation method. (a) Pyrex glass cell, (b) Thermostat and water motor, (c) Potentiostat and voltage sweep generator, (d) Recorder.

immersed in the 2% CHX solution had corrosion which did not cause a significant change in the surface of the file, and was similar to the control sample (Figure 3). The SEM image of the file in contact with the 2.5% NaOCl solution showed corrosion zones and residual products (Figure 4). Highly dense corrosion zones and residual products which were spread over a large area on the surfaces of files were observed in the SEM images of the instruments immersed in the 5% NaOCl (Figure 5) and 15% EDTA solutions (Figure 6).

DISCUSSION

Endodontic instruments are always subject to stress and corrosive environments in root canal treatment, and during the cleaning and shaping processes in endodontic therapy. The breakage of files while performing the chemical and mechanical debridment of root canals can adversely affect the result of the endodontic treatment. Metal fatigue has been considered as a primary factor in Ni-Ti instrument fracture in clinical conditions.[10] However, the breakage of files should not only be attributed to metal fatigue but also to the negative effects of corrosion on the fracture resistance of files. Metallic surfaces are affected by corrosion which leads to pitting and porosity, which results in the reduced cutting ability of endodontic files. A prolonged use of these files with a reduced cutting ability may increase metal fatigue and the risk of plastic deformation/intracanal separation.[1,10] In this study, the ProTaper Universal Ni-Ti file system, which is widely used for the cleaning and shaping procedures of root canals, was evaluated in terms of its corrosion rate.

Table 1. Corrosion rate (i_{cor}) values of Ni-Ti rotary files in tested solutions

Irrigation solutions	Mean ± SD	Median
2% CHX	0.1214±0.0504	0.12
5% NaOCl	3.150±1.3246	3.25
2.5% NaOCl	0.4529±0.0897	0.45
15% EDTA	2.100±0.2449	2.10

Ni-Ti: Nickel-titanium, SD: Standard deviation, CHX: Chlorhexidine gluconate, NaOCl: Sodium hypochlorite, EDTA: Ethylenediaminetetraacetic acid.

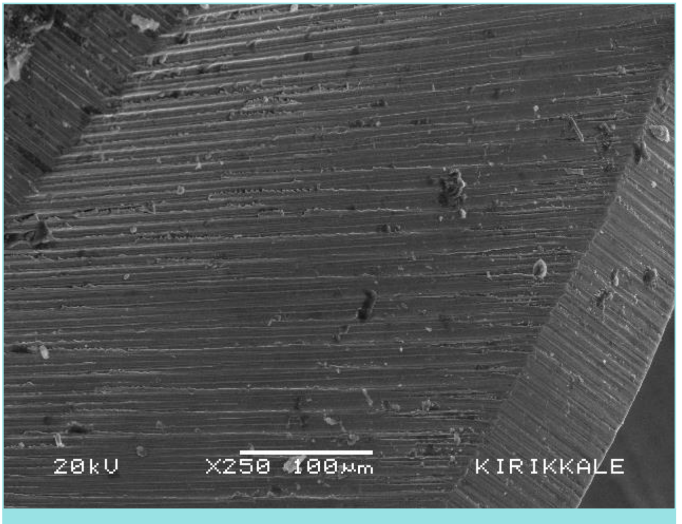

Figure 2. SEM of untreated Ni-Ti control file revealed no evidence of corrosion (x250).

SEM: Scanning electron microscope, Ni-Ti: Nickel-titanium.

Various methods, including atomic force microscopy, the linear polarization method, the alternating current impedance method, open circuit potential and energy dispersive X-ray microanalysis, are used to determine corrosion rates of metals and alloys. In this research, the Tafel extrapolation method was used as an electrochemical test in order to determine the corrosion features of the metals and alloys. Electrochemical techniques which are based on polarization-resistance techniques and polarization profiles are reliable and efficient methods to investigate the corrosion mechanisms of endodontic files. These methods are also used to evaluate the

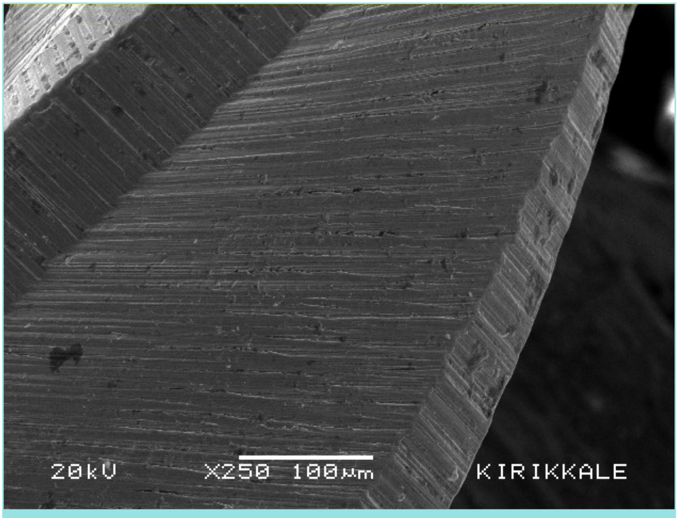

Figure 3. SEM of 2% CHX group revealed the lowest corrosion area (x250).

SEM: Scanning electron microscope, CHX: Chlorhexidine gluconate.

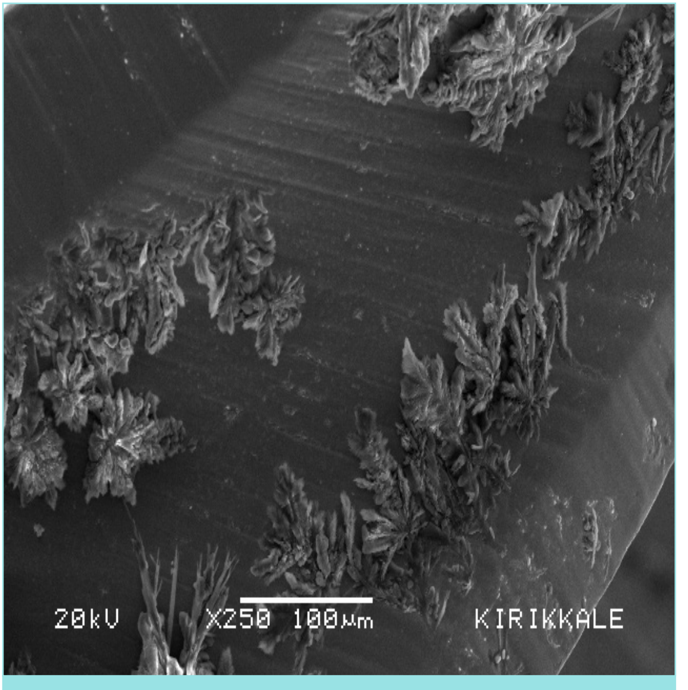

Figure 4. SEM observed that corrosion occurred in local regions in 2.5% NaOCl group (x250).

SEM: Scanning electron microscope, NaOCl: Sodium hypochlorite.

corrosion sensitivity of recently created and enhanced materials for the production of instruments.[1,8,9]

In the present study, the corrosion rates of Ni-Ti instruments in four various irrigants from the strongest to the mildest were as follows: 5% NaOCl >15% EDTA >2.5% NaOCl >2% CHX. These results are likely to be related to active Cl^- which is an important aggressive ion which usually causes increased corrosion rates as NaOCl contains active chloride (Cl^-) ions.[11] The corrosion rate was lowest in the CHX group, which can be explained by the fact that CHX does not contain active Cl^- ions.

Complexes are formed by EDTA with metal ions (e.g., Co, Cr, Fe, Ni, etc.) at low pH (<4).[6] The capability of EDTA to passivate and ensure the protection of instruments is explained by its capability to form complexes with iron in order to create an inhibiting barrier to oxidation and corrosion.[12,13] Furthermore, it is very difficult for the large molecules of EDTA to concentrate and orient to the pit in order to increase the acidity to sufficient values for the purpose of triggering corrosion.[14] However, EDTA easily forms Ni-Ti complex in the range of pH 5.0-8.0, and this complex dissolves abundantly in polar solvents so that the dissolution of the complex leads to an increase in the corrosion rate.[15] Hence, 15% EDTA was found to have the second highest corrosion rate following 5% NaOCl solution, and its corrosion layer was seen to be diffused throughout the entire surface of the file in this research.

The corrosion rate of the 2.5% NaOCl group was lower than that of the 5% NaOCl group due to the decreased the amount of active Cl^- ions by concentration. O'Hoy et al.[8] found that Milton's solution (19% NaCl + 1% NaOCl) was more corrosive than a 1% NaOCl solution. Since 2.5% NaOCl solution exhibited less corrosion on the surface of the files, it may be safer to use it as an irrigant while using rotary Ni-Ti files.

Darabara et al.[16] found that H-files manufactured from two various stainless steel alloys and one file manufactured from Ni-Ti alloy were not susceptible to pitting or crevice corrosion in NaOCl and R-EDTA solutions. In contrast with our findings, they showed that all materials demonstrated higher corrosion potential in NaOCl compared with R-EDTA. Cavalleri et al.[17] also reported that contact with NaOCl for up to 10 minutes does not alter the surface of ProTaper Universal files through corrosion. This result may be attributed to the difference in their analysis method from ours. However, Dartar et al.[9] compared the corrosion rates of stainless steel endodontic files placed in various irrigation solutions and found that 0.2% CHX, 5.25% NaOCl and chlorinated soda with potassium hydroxide led to significant corrosion on the surface of the chosen stainless steel files. The lowest corrosion rate of stainless steel files was seen in 17% EDTA solution.[8] Yum et al.[18] compared the corrosion tendency of used and unused ProTaper Universal Ni-Ti files and different immersion temperatures of NaOCl. They demonstrated that the solution temperature and the chloride ion concentration had an effect on the passivity and corrosion resistance of Ni-Ti files after clinical use. Anto et al.[19] concluded that 2.5%, 5.25% and 8.25% NaOCl caused a deterioration on the ProTaper Universal Ni-Ti files surface. In our study, we also demonstrated that NaOCl at higher concentrations caused highly dense corrosion zones on the Ni-Ti file surface. In accordance with our findings, Sağlam et al.[20] found that 2% chlorhexidine demonstrated limited surface alterations on ProTaper Universal Ni-Ti files when compared to the 2.5% NaOCl, 5% NaOCl and a mixture of tetracycline, citric acid, and detergent (MTAD). Several studies also observed localized corrosion defects

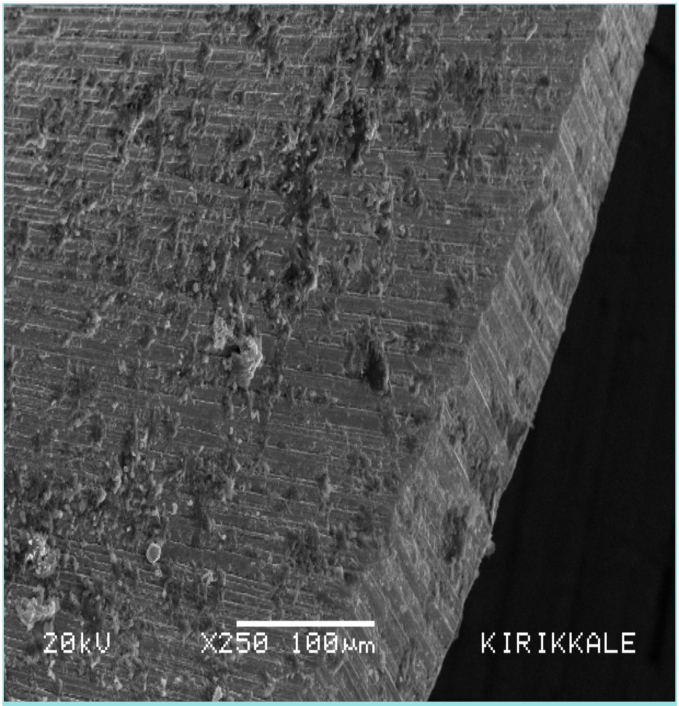

Figure 5. SEM images of a compact and complex corrosion layer on files which were immersed in 5% NaOCl (x250).

SEM: Scanning electron microscope, NaOCl: Sodium hypochlorite.

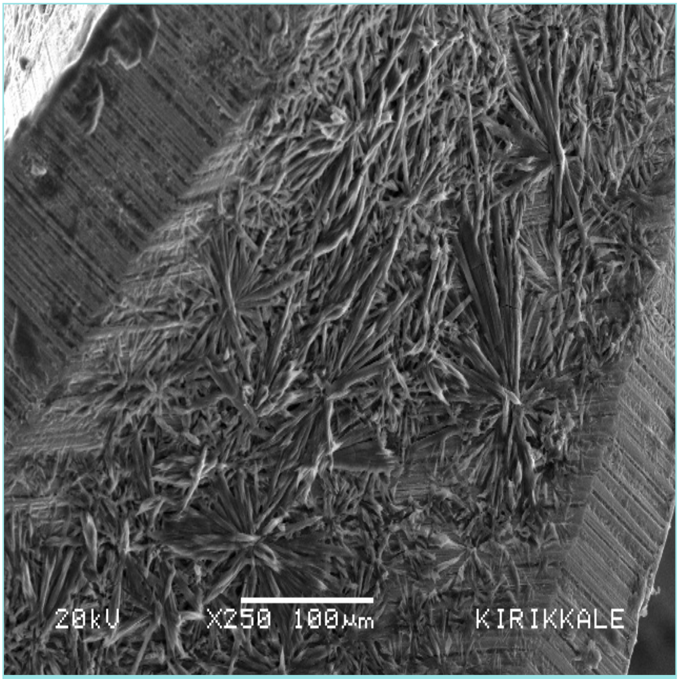

Figure 6. SEM images of a compact and complex corrosion layer on files which were immersed in 15% EDTA (x250).

SEM: Scanning electron microscope, EDTA: Ethylenediaminetetraacetic acid.

and micro-cracks on the surfaces of ProTaper Universal Ni-Ti files after immersion in 2.5 and 5% NaOCl solution.[2,6,9,16]

Berutti et al.[21] showed the presence of localized corrosion attack on the surface of ProTaper Ni-Ti files which were immersed in 5% NaOCl via SEM images. In our study, large corroded areas on the surfaces of ProTaper Ni-Ti files immersed in 5% NaOCl solution were observed in SEM images. Cassol et al.[22] also noted uniform corrosion areas on ProTaper Universal and WaveOne Gold Ni-Ti files which were immersed in NaOCl via SEM imaging.

Study Limitations

The major limitation of the present study was that it was only carried out with Protaper Universal Ni-Ti files, whereas numerous different types of file systems have been introduced to the dental market over the previous few years. Manufacturers are increasingly trying to develop improved Ni-Ti rotary instruments aimed at providing better mechanical and metallurgical properties, enhanced cutting efficiency, fatigue resistance, and corrosion resistance. Cryogenic treatment, thermal nitridation, ion implantation and electro-polishing are a few examples of the strategies used to create enhanced instrument surfaces. There is no doubt that different instruments manufactured in different methods can have different properties in terms of their corrosion resistance. Future studies on the current topic are therefore recommended in order to reveal the corrosion behaviors of newly developed Ni-Ti rotary instruments. Similarly, in future investigations, it would be interesting to conduct research with different types of newly developed endodontic irrigants in order to disclose their corrosion potentials on endodontic instruments. One of the other limitations of the present study was that the *in vitro* conditions may not reflect the *in vivo* conditions accurately since dentin has considerable buffering capacity against irrigants. Therefore, further studies should be conducted in order to evaluate the corrosion rates of root canal irrigants during root canal treatment procedures with Ni-Ti rotary files under *in vivo* conditions.

CONCLUSION

Understanding the corrosion characteristics of endodontic files in various irrigation solutions is essential because of the corrosion tendencies of files in the course of root canal preparation may cause instrument fractures, which are unpleasant accidents. In conclusion, clinicians should be aware of defects which may form on files and the optimal number of uses of each root canal instrument in order to reduce the risk of instrument fracture. In terms of the tested solutions in the present study, the least damaging and the safest irrigation solution for these instruments is CHX.

MAIN POINTS

- The average corrosion rates of the files in the four various irrigation solutions tested are as follows: 5% NaOCl >15% EDTA >2.5% NaOCl >2% CHX.
- According to the corrosion rates determined, the rate of corrosion increases as the concentration of NaOCl solution increases.
- In the SEM investigation, highly dense corrosion zones and corrosion residual products were found on the surfaces of the file selected from the 5% NaOCl solution group.
- The 2% CHX solution used in our study had the lowest average corrosion rate on Ni-Ti rotary files. The corrosive effect of this solution on nickel-titanium files is minimal and so was preferential, as determined via the SEM images obtained.
- The average corrosion rates determined by the Tafel extrapolation method in the four various irrigation solutions used were supported by the SEM findings.

ETHICS

Ethics Committee Approval: Ethics committee approval was not required.

Informed Consent: Informed consent approval was not required.

Peer-review: Externally peer-reviewed.

Authorship Contributions

Concept: T.Ö., B.S., M.D.Ö., F.B., U.A., Design: T.Ö., B.S., M.D.Ö., Supervision: T.Ö., B.S., M.D.Ö., Fundings: T.Ö., B.S., M.D.Ö., Materials: T.Ö., B.S., M.D.Ö., F.B., U.A., Data Collection and/or Processing: T.Ö., B.S., F.B., U.A., Analysis and/or Interpretation: T.Ö., B.S., F.B., U.A., Literature Search: F.B., U.A., Writing: F.B., U.A., Critical Review: F.B., U.A.

DISCLOSURES

Conflict of Interest: No conflict of interest was declared by the authors.

Financial Disclosure: The authors declared that this study had received no financial support.

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