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Research Article

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Corrosion resistance assessment of nickel-titanium endodontic files with and without heat treatment

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Conflict of Interest
No potential conflict of interest relevant to this article was reported.

Author Contributions
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ABSTRACT

Objectives: The aim of this study was to evaluate the corrosion resistance of heat-treated (Reciproc and WaveOne) and non-heat-treated (ProTaper and Mtwo) superelastic nickel-titanium endodontic files when immersed in a 5.25% sodium hypochlorite solution.
Materials and Methods: Anodic polarization curves were obtained with potential sweeps that began at the open circuit potential or corrosion potential (E_{corr}). The pitting potential (E_{pit}) was identified on the anodic polarization curve as the potential at which a sudden increase in current was observed. The micromorphology of the 28 tested files was analyzed before and after the electrochemical assay using scanning electron microscope (SEM). The data were analyzed using 1-way analysis of variance with the *post hoc* Bonferroni test (for E_{corr}) and the Student *t*-test for independent samples (for E_{pit}).
Results: The mean E_{corr} values were 0.506 V for ProTaper, 0.348 V for Mtwo, 0.542 V for Reciproc, and 0.321 V for WaveOne files. Only WaveOne and Protaper files exhibited pitting corrosion, with E_{pit} values of 0.879 V and 0.904 V, respectively. On the SEM images of the ProTaper and WaveOne files, cavities suggestive of pitting corrosion were detected.
Conclusions: Signs of corrosion were observed in both heat-treated and non-heat-treated files. Of the evaluated files, WaveOne (a heat-treated file) and ProTaper (a non-heat-treated file) exhibited the lowest corrosion resistance.

Keywords: Corrosion; Endodontics; Root Canal

INTRODUCTION

The main objective of the biomechanical preparation of root canals is to remove the pulp tissue and its necrotic debris. The clean result, shape, and dentin permeability that can result from this process promote hermetic obturation. To achieve this, a combination of irrigation and mechanical instrumentation of the root canal system is applied. This process involves the use of appropriate files, reamers, milling cutters, ultrasonic instruments or other mechanical devices, and rotary systems with nickel-titanium (NiTi) files.

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Root canal instruments composed of a NiTi alloy were introduced in 1988 to overcome the rigidity of materials manufactured out of stainless steel [1]. The use of very flexible endodontic instruments made of superelastic NiTi alloys transformed the methods available for root canal instrumentation, reducing procedural errors and enhancing security and predictability [2]. Endodontic treatment has since benefited from the development of NiTi instruments because their increased taper allows for simpler and more efficient root canal preparation. Compared to those made from stainless steel alloys, tools produced from NiTi alloys have advantageous mechanical properties, such as greater resistance to fracture caused by twisting clockwise or counterclockwise, greater fatigue resistance, and high flexibility [3-5]. This phenomenon is mainly due to 2 properties inherent to NiTi alloys: the shape memory effect and superelasticity [3].

In clinical practice, endodontic files are constantly exposed to auxiliary chemicals that are used to disinfect root canals. One of the most commonly used of these chemicals is sodium hypochlorite (NaOCl). NaOCl solution is used inside the root canal to dissolve organic matter. This chemical also acts as a bactericide and operates in conjunction with the endodontic files to chemo-mechanically clean and prepare the root canal [6]. Contact with these substances may degrade the surfaces of the endodontic files and cause alloy corrosion, enabling the formation of file fractures in the root canal [7]. Thus, the corrosion of endodontic files is relevant to several areas of practice due to the potential for negative consequences for both the professional and the patient [8-14].

Endodontic instruments composed of NiTi present a high risk of separation due to torsional or flexural fatigue (cyclic fatigue). Chemo-mechanical root canal preparation, file cleaning, and chemical disinfection and sterilization corrode the endodontic instruments, which can weaken them and reduce their fracture resistance [15]. To increase the resistance to torsional fracture by enhancing the torsional flexibility of NiTi instruments, manufacturers and researchers have designed a special thermomechanical process that enables the fabrication of a new alloy, termed M-Wire [16,17].

In this context, we aimed to evaluate the corrosion resistance of heat-treated (Reciproc and WaveOne) and non-heat-treated (ProTaper and Mtwo) superelastic NiTi endodontic files when immersed in a 5.25% NaOCl solution.

MATERIALS AND METHODS

Twenty-eight 25-mm-long NiTi files were tested and divided into 4 groups as follows. Group 1 included ProTaper files Universal (Dentsply-Maillefer, Ballaigues, Switzerland), group 2 included Mtwo files (VDW, Munich, Germany), group 3 consisted of Reciproc R25 files (VDW), and group 4 consisted of WaveOne files (Dentsply-Maillefer) (**Figure 1**).

The corrosion resistance was evaluated using the potentiodynamic polarization test with a potentiostat (PGSTAT 128N, Metrohm Autolab BV, Utrecht, The Netherlands) coupled to a computer with software (NOVA 2.0, Metrohm Autolab BV) that utilized a potential sweep to produce results shown as anodic polarization curves. The electrochemical tests were performed using the classical 3-electrode scheme, with these electrodes including a saturated calomel electrode as a reference, a large-area platinum electrode as the counter-electrode, and the working electrode.

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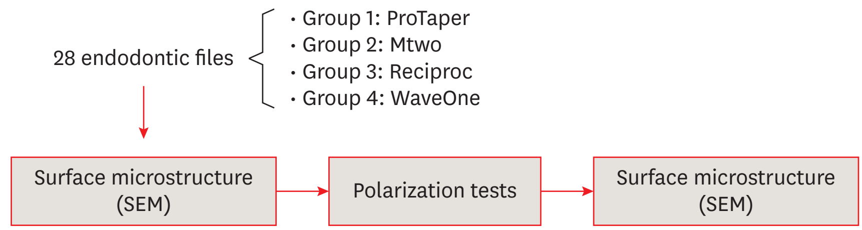

Figure 1. Representative diagram of the experimental design.
SEM, scanning electron microscope.

Before conducting the electrochemical tests, the files were cleaned in an ultrasonic tank (LS-0, 8D LimpSonic, São Paulo, Brazil) in an acetone solution for 30 minutes. The working electrode consisted of files coupled to a system designed to make a connection with a copper wire. This copper wire had been laminated at one end in a refiner mill until it was 1 mm thick, and it was bent to receive the wire cable. A device specially designed for the fixing of the metallic contact file and copper wire was installed and tightened. The brass connection was then covered with silicone to isolate its contact with the working solution. The area exposed to the 5.25% NaOCl solution was 0.04 cm^2. To help stabilize the electric current, the platinum connector was attached to the copper wire and wrapped in Teflon tape to maintain proximity to the working and platinum electrodes. To standardize the distance between the working and platinum electrodes, a 0.5-mm latex rubber template was used.

To determine the circuit potential, tests of the electrochemical potential were implemented after the samples had been immersed for 60 minutes in the 5.25% NaOCl solution (pH 6.5) at 37°C ± 1°C. In each assay, 100 mL of the tested solution was used and then replaced. Before the test, the pH of the solution was measured at 6.5 with a digital pH meter (HI8314, Hanna Instruments, Woonsocket, RI, USA), and the temperature was stabilized at 37°C using a magnetic stirrer with temperature control (78 HW-1, Biomixer, Ribeirão Preto, Brazil).

The anodic polarization curves were obtained with potential sweeps that began from the open circuit potential or corrosion potential (E_{corr}) of each file at a rate of 0.333 mV/sec. Before the experiment, the electrodes were kept in the solution so that electrochemical equilibrium could be achieved, and the value of the open circuit potential was set. The corrosion resistance of the files was determined by the pitting potential (E_{pit}), identified on the anodic polarization curve as the potential at which the current suddenly increased.

The entire experiment was conducted within a Faraday cage, with the goals of isolating the system from external electromagnetic waves, avoiding interference, and improving the reliability of the results. The surface microstructure of the files was visually assessed using a scanning electron microscope (SEM) (LEICA/LEO Stereoscan S440, Electron Microscope Unit, Rondebosch, South Africa) equipped with a retro-spread electron detector and secondary electron detector. The files were observed under the SEM before and after polarization testing.

To compare the groups, 1-way analysis of variance with the *post hoc* Bonferroni test (E_{corr}) and the Student *t*-test for independent samples (E_{pit}) was used. For data analysis, SPSS version 23 software (SPSS, Armonk, NY, USA) was used, and the significance level was set at $p < 0.05$.

RESULTS

Polarization tests

The E_{corr} values of the 4 groups of files are displayed and compared in **Table 1**. The mean E_{pit} of the ProTaper (non-heat-treated) files was significantly higher than that of the WaveOne (heat-treated) files (**Table 1**) ($p < 0.05$). A significant difference was observed with regard to E_{corr} among all groups; the Bonferroni test was then used to test relationships between the file groups in pairs (**Table 2**), and all of the pairs of groups presented significant differences.

Polarization curves obtained for the Reciproc (**Figure 2**) and Mtwo (**Figure 3**) files showed no indications of pitting corrosion. In contrast, the ProTaper (**Figure 4**) and WaveOne (**Figure 5**) files exhibited a sudden increase in current, measured as E_{pit} and characterized by a horizontal plateau on the chart, suggesting the occurrence of pitting corrosion.

Table 1. Mean ± standard deviation (SD) of the corrosion potential (E_{corr}) and pitting potential (E_{pit}) values of ProTaper, Mtwo, Reciproc, and WaveOne files

Files	E_{corr}*	E_{pit}†
Heat-treated		
ProTaper	0.506 ± 0.013	0.904 ± 0.010
Mtwo	0.348 ± 0.006	-
Non-heat-treated		
Reciproc	0.542 ± 0.001	-
WaveOne	0.321 ± 0.001	0.879 ± 0.005
p value	< 0.001*	0.004†

Values are presented as mean ± SD. Data were analyzed using analysis of variance.
*Comparison between 4 groups; †Comparison between 2 groups.

Table 2. A *p* values for the comparisons of corrosion potential (E_{corr}) between ProTaper, Mtwo, Reciproc, and WaveOne files

Comparisons of E_{corr}	ProTaper	Mtwo	Reciproc	WaveOne
ProTaper	-	< 0.05	< 0.05	< 0.05
Mtwo	< 0.05	-	< 0.05	< 0.05
Reciproc	< 0.05	< 0.05	-	< 0.05
WaveOne	< 0.05	< 0.05	< 0.05	-

Data were analyzed using the *post hoc* Bonferroni test.

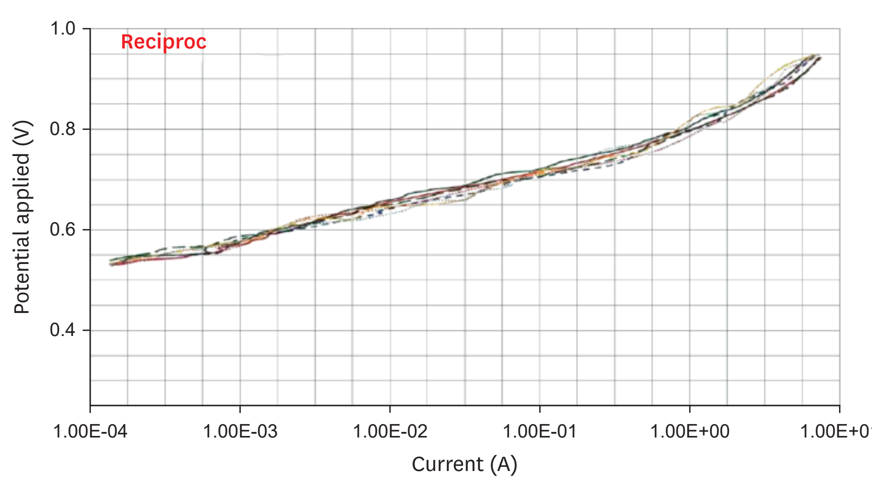

Figure 2. Anodic polarization curves of the Reciproc files in 5.25% sodium hypochlorite (NaOCl) solution.

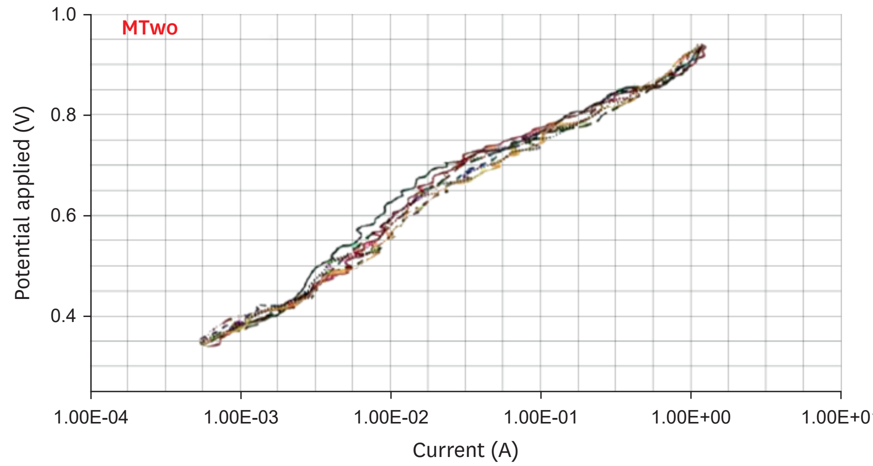

Figure 3. Anodic polarization curves of the Mtwo files in 5.25% sodium hypochlorite (NaOCl) solution.

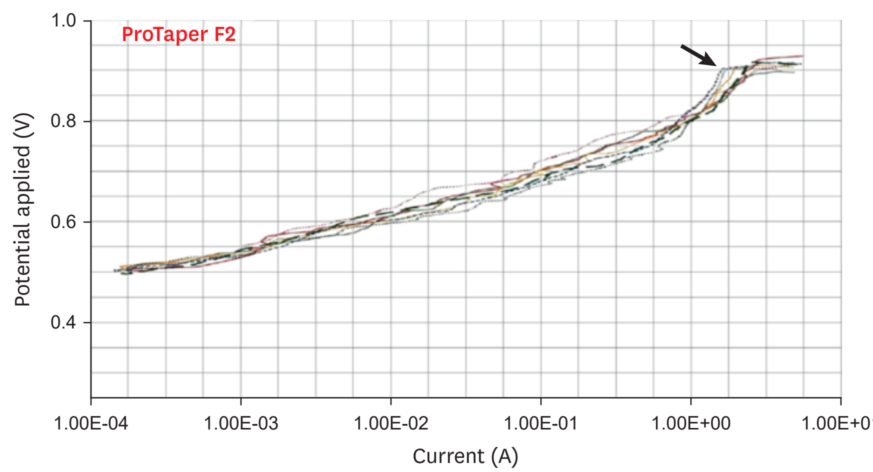

Figure 4. Anodic polarization curves of the ProTaper files in 5.25% sodium hypochlorite (NaOCl) solution. The arrow indicates the cavity of pitting.

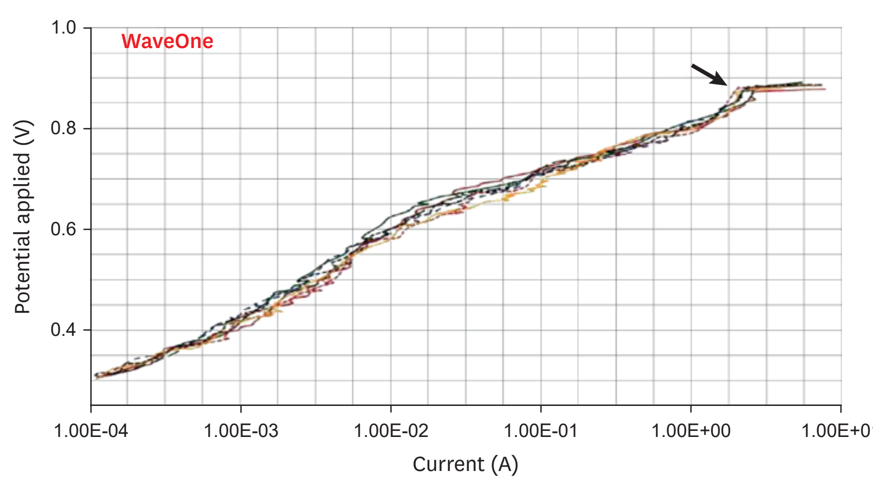

Figure 5. Anodic polarization curves of the Mtwo files in 5.25% sodium hypochlorite (NaOCl) solution. The arrow indicates the cavity of pitting.

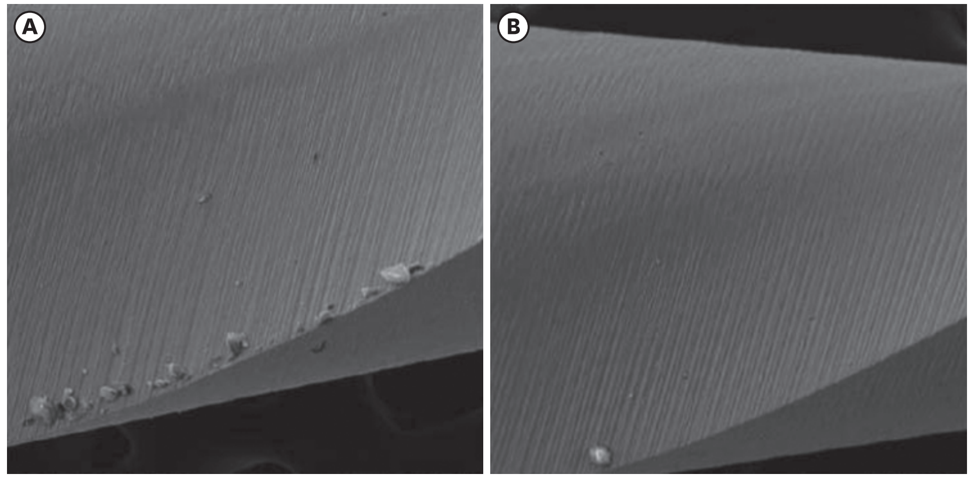

Figure 6. Scanning electron microscopic image of Reciproc files. (A) Before (×304) and (B) after the electrochemical test (×306).

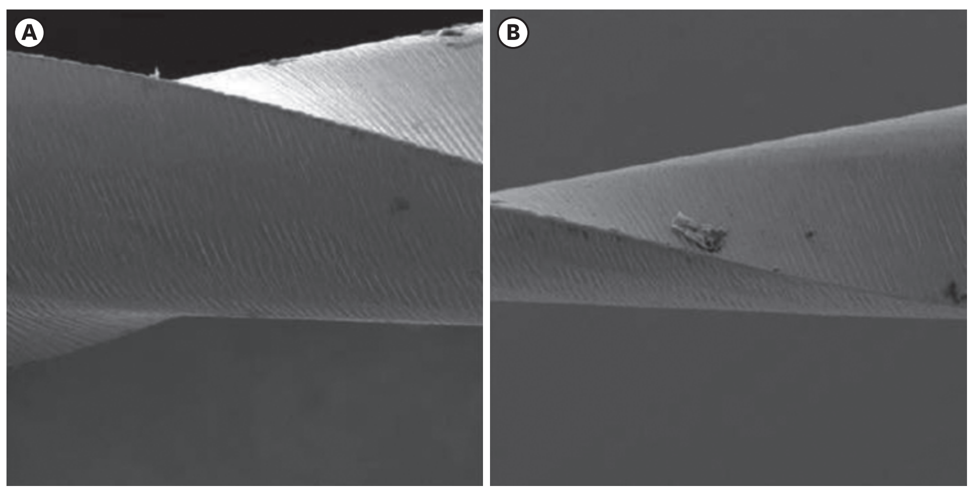

Figure 7. Scanning electron microscopic image of Mtwo files. (A) Before (×296) and (B) after the electrochemical test (×298).

Surface microstructure

SEM images of the surfaces of the Reciproc (**Figure 6**) and Mtwo (**Figure 7**) files obtained before and after the cyclic polarization test showed regular surfaces with adhered particles. In the SEM images after the cyclic polarization of the ProTaper (**Figure 8**) and WaveOne (**Figure 9**) files, cavities suggestive of pitting corrosion were detected and confirmed the results obtained from the polarization curves.

DISCUSSION

Irrigation solutions can have a corrosive and progressively damaging effect on endodontic instruments during root canal preparation [7]. To increase the fracture resistance of the endodontic files, some changes have been made to the NiTi alloy microstructure and the overall file structure. Heat treatment, applied by uniformly heating the alloy to 500°C and then rapidly cooling it, is intended to optimize the mechanical properties and particularly the fracture resistance of the alloy [2]. The heat-treated M-Wire alloy is composed of structures

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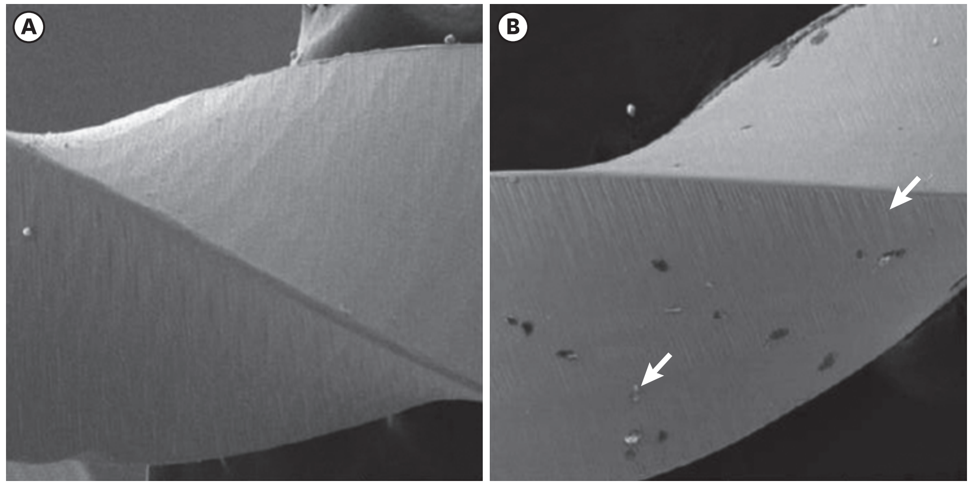

Figure 8. Scanning electron microscopic image of ProTaper files. (A) Before (×256) and (B) after the electrochemical test, with the presence of corrosion by pitting (×250).

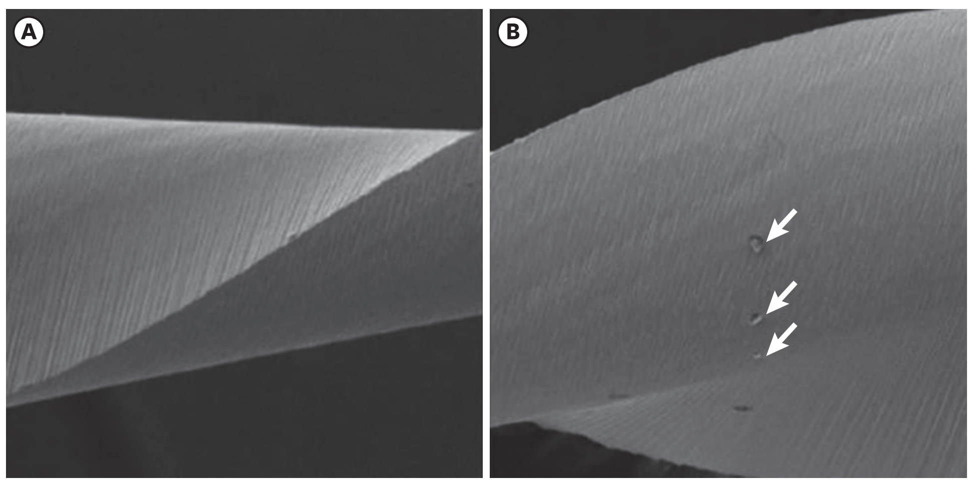

Figure 9. Scanning electron microscopic image of WaveOne files. (A) Before (×304) and (B) after the electrochemical test, with the presence of corrosion by pitting (×306).

in the R-phase, or rhombohedral phase, which is associated with high shape memory and superelasticity. Moreover, the Young modulus of the R-phase is lower than that of austenite. Therefore, tools that are composed of materials in the R-phase will be more flexible than tools that are not [2]. Due to the presence of some martensite that does not transform into austenite, the alloy features a more torsion-resistant microstructure than traditional NiTi, which contains only austenite crystals [2,18].

In general, E_{corr} is an indicator of the ionization tendency of a material in a specific medium [7]. In this study, the average E_{corr} values for the 4 studied groups of files ranged from 0.321 V to 0.506 V, resembling a result obtained by Darabara *et al.* [4] in which the values ranged from 0.350 V to 0.420 V for stainless steel and NiTi endodontic instruments. In the present study, the WaveOne group had the lowest mean E_{corr} value (0.321 V), meaning that the files of this group had the greatest tendency to corrode. This was expected, as this alloy had not been heat-treated. Moreover, after reviewing the findings published by Talha *et al.* [5] and the research carried out by Lee and Yoon [10], it is clear that titanium files have a relatively low tendency to corrode, as the E_{corr} value of the 316LN stainless steel alloy, which does not

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contain titanium, in NaCl solution is −0.5 V, a value approximately 70% lower than the E_{corr} value found in this study.

Endodontic files are in constant contact with irrigating solutions during the cleaning and shaping of the root canal [19]. Even short-term contact with these solutions can cause changes in the surfaces of NiTi instruments; these include the development of pitting corrosion spots, which can lead to file fractures in the root canal [8]. In addition, a NiTi alloy can act as an anode and is therefore susceptible to corrosion [14]. Understanding the corrosion resistance of a material in a given environment is important in reducing the occurrence of possible failures, including fractures [11].

Pitting corrosion is a type of localized corrosion in which an aggressive environment breaks through the protective layer and progress more deeply into the material [20-22]. Corrosion resistance is related to the protective capacity of the passivation layer in supporting external aggressions until it breaks down and leaves the alloy susceptible to corrosive attack [9]. In the present study, the E_{pit} varied from 0.879 V to 0.904 V; these were lower values than those obtained in the study by Darabara *et al.* [4] (around 1.2 V). This difference may be associated with the pH of the solution used; in Darabara *et al.*, [4] the pH of the solution was 10.6, whereas in the present study, the pH was 6.5. The lower E_{pit} value observed in this study can be justified by the fact that a low pH favors corrosive processes. In another study [9], NiTi files subjected to an electropolishing surface treatment exhibited an E_{pit} of 0.6 V in NaCl solution, a lower value than those found in this study. Therefore, we can infer that the electropolishing surface treatment does not directly help maintain the protective layer, as shown by the pitting potential above 0.6 V.

The pitting corrosion (or lack thereof) of the evaluated files may be more closely related to the design of the endodontic file than to the use of heat treatment. The ProTaper and WaveOne files, which exhibited corrosion, are more irregular in their design than the other files, with variations in the section along the axis of the instrument. The cross-section of a ProTaper file is convex triangular, whereas that of a WaveOne file is modified triangular (apical) and convex triangular (coronal) [2]. In contrast, the files that showed no corrosion—Mtwo (non-heat-treated) and Reciproc (heat-treated)—have S-shaped cross-sectional designs [2]. This aligns with the principles of enthalpy and entropy, since the energy released by chemical reactions (enthalpy) and the extent of molecular disorganization according to the contact area (entropy) are higher in surfaces with major irregularities, directly influencing the corrosion resistance of the material [23,24]. As such, the triangular format appears to be associated with higher levels of enthalpy and entropy, favoring corrosive processes relative to S-shaped files.

According to Souni *et al.* [25], the factors that influence corrosion are the chemical composition (levels of Ni, Ti, and alloy impurities), the degree of microstructural homogeneity, and the presence of residues on the surface of the material. In the present study, the surface microstructure analysis conducted via SEM demonstrated that both the files from the Reciproc group and the files from the Mtwo group included particles adhered to a regular surface without indicators of corrosion, similar to SEM images obtained by Thompson [26]. However, the surface irregularities (milling marks) and metal flash (roll-over) on the cutting edges may compromise the cutting capacity of these instruments and potentially foster corrosion [26]. The Reciproc and Mtwo files differed from the ProTaper and WaveOne files, which exhibited superficial alterations on SEM with cavities suggestive of pitting. The SEM findings were confirmed by the cyclic polarization results, in which both groups displayed significant changes in the electric current at a certain potential, which was considered to represent E_{pit}. A surface morphology indicative of pitting was observed in the ProTaper and WaveOne groups, and the SEM images were similar to those obtained in another study (conducted by Bonaccorso *et al.* [9]) that also detected the same type of corrosion in NiTi files.

CONCLUSIONS

Signs of corrosion were observed in both heat-treated and non-heat-treated files. WaveOne (heat-treated) and ProTaper (non-heat-treated) files presented lower corrosion resistance than the other evaluated files.

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