BASIC RESEARCH – TECHNOLOGY

Electrochemical Properties of Nickel-Titanium Rotary Endodontic Instruments

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ABSTRACT

Introduction: Nickel-titanium rotary endodontic files have been commercially available for decades, but more recent innovations have introduced heat-treated and surface-treated files. This study investigated the corrosion properties of various nickel-titanium files in normal saline and sodium hypochlorite (NaOCl). **Methods:** Ten different file brands of size 40 with a 0.04 taper were subjected to electrochemical testing in 0.9% NaCl (saline) and 5.25% NaOCl at room temperature. The Open Circuit Potential (OCP) was observed for 1 hour followed by a cyclic polarization test from −300 to 700 mV and back to −300 mV (vs OCP). Nonparametric ANOVA and a pairwise comparison ($P < .05$) were used for statistical analysis of the OCP at 1 hour and the corrosion current (Icorr) obtained via the cyclic polarization test. **Results:** Significant differences ($P < .05$) were found between files with respect to OCP and Icorr in both solutions. Nine files exhibited significantly greater ($P < .05$) Icorrs in NaOCl than in saline. Conversely, pitting corrosion was observed in the saline solution but not NaOCl. Weak and/or moderate correlations existed between OCP and Icorr measures in the 2 solutions. **Conclusion:** Significant differences in electrochemical properties were observed among the 10 brands of files. Overall, there was not a clear trend between conventional, heat-treated, or surface-treated files among OCP or Icorr in either solution. *(J Endod 2024;50:1143–1150.)*

KEY WORDS

Corrosion current; endodontic rotary files; nickel-titanium; saline; sodium hypochlorite

SIGNIFICANCE

Corrosion of endodontic files may have a negative, conjoint influence on their cyclic flexural fatigue lifetime. Similarly, in the event of file separation, file segments entombed within a canal will release ions via corrosion that may have biological consequences. This research showed differences in the electrochemical properties of 10 rotary NiTi files. Pitting corrosion was observed in saline but not sodium hypochlorite.

Root canal treatment aims to salvage a tooth by thorough chemo-mechanical debridement of infected or necrotic pulp tissue and sealing the root canal system with gutta percha, sealer, and a good coronal restoration. The process involves the application of suitable hand files, reamers, nickel-titanium (NiTi) rotary files, and ultrasonic instruments with concomitant use of irrigation solutions. Traditionally, canal preparations were prepared using stainless steel files, but its potential for ledges, transportation, and perforations were lessened with the advent of NiTi files[1,2]. NiTi alloy was first introduced in the 1960s by William Bueller at the United States Naval Ordnance Laboratory, and NiTi files were proposed by Walia et al[1] for endodontic treatment in 1988 due to NiTi's greater flexibility compared to stainless steel files. In the early 1990s, the first commercially available NiTi files were marketed[3] and since then they have revolutionized endodontic practice by enhancing predictability and lessening procedural errors. Nevertheless, file separation or fracture remains a possibility with NiTi files[4]. The 2 most common mechanisms with which endodontic files fail when rotating in the canal are cyclic flexural fatigue and torsional overload fracture[5]. In addition, processing defects like surface irregularities may play a role in clinical failures[6,7]. These defects likely result from the typical machine grinding necessary to create the cutting edges of most NiTi files and may act as stress concentration and crack initiation points[8–10].

Since their inception, NiTi endodontic files have undergone numerous innovations in design and/or manufacturing with a goal of improving mechanical properties, cutting ability, canal shaping, cleaning efficacy, and/or clinical performance[11]. For all NiTi files, different phases/crystal structures (austenite, martensite, and rhombohedral/R-phase) may be present at oral temperature depending on the manufacturing and heat-treatment parameters. In the current research, 10 endodontic rotary NiTi files were evaluated and are described here in more detail. ProFile, K3, and EndoSequence may be categorized as austenitic NiTi, capable of exhibiting superelasticity[12]. Twisted Files exhibit superelasticity as well via having an austenite finish (Af) temperature at or below room temperature[13,14], but it is twisted to its final shape rather than the grinding of traditional NiTi files. K3XF is descended from the K3 file, but is

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processed with a thermal treatment that slightly elevates its austenite finish temperature compared to K3[15], yet it still remains superelastic in use. ProFile Vortex is often described as being "M-wire" due to some retained, stabilized martensitic structure, though it too behaves superelastically[13,16]. EndoSequence CM, HyFlex CM, HyFlex EDM, and Vortex Blue are heat-treated postprocessing, predominantly are martensitic, and exhibit thermal shape memory properties[13]. Among the above files, those with surface alterations are the electropolished EndoSequence and the electrical discharge machined HyFlex EDM[3].

In clinical practice, NiTi files are exposed to chemicals that are used to treat the canals. One of the key steps in root canal treatment is the use of irrigating solutions after each mechanical instrumentation, with 5.25% sodium hypochlorite (NaOCl) commonly used[17,18]. NaOCl is used as an endodontic irrigant mainly because of its bactericidal, tissue-dissolving capabilities, and easy penetration to the root canals due to its low viscosity[19]. However, it is toxic to vital tissues and is corrosive to metals[19]. Several studies have examined how exposure of endodontic files to NaOCl affects their surface, with some, but not all, showing general corrosion or pitting corrosion[20–25]. It is probable that these environmentally induced defects (pitting) could contribute to file separation via acting as stress concentrators, as confirmed by Cheung and Darvell[26]. Further, any existing surface defects like cracks may experience chemically-assisted, accelerated crack growth that lowers the fatigue resistance of the NiTi files. Similarly, many studies and/or brands of files, but not all, show a decrease in cyclic flexural fatigue life when endodontic NiTi files are exposed to NaOCl[22,24,25,27–33]. It is possible the disparity in corrosion amount and effect on fatigue properties when NiTi files are exposed to NaOCl could be file specific; however electrochemical testing of NiTi files is not prevalent, though some investigations have occurred[34–36].

In the event of file separation in the canal, retrieval of the fractured segment is desirable for long-term clinical success, but it is not always possible[37]. Sealing in the fractured segment may still occur, but the extent of leakage has been shown to be highly variable in an *in vitro* study[38]. In such instances, the non-retrieved file may be exposed to biological fluids (eg, dentinal or periapical fluid), triggering corrosion and release of ions to surrounding tissue. Though the release of ions from a file segment left in a canal would be far less than that from the intentional full dissolution of a file, both soluble and insoluble NiTi dissolution products have been shown to be toxic to periodontal ligament cells[39]. In the present study, saline was used as a simulated body fluid in investigating the corrosion of NiTi files as if the file segment remained in the canal.

With consideration to the lack of information on the electrochemical properties of modern NiTi endodontic files in relevant solutions, this study aimed to study the corrosion properties of various endodontic files, including conventional, heat-treated and surface-treated NiTi files, when exposed to 5.25% NaOCl and saline solution. The measured properties include: The open circuit potential (OCP) and corrosion current (Icorr). The OCP is also known as the rest potential or equilibrium potential and depends on the environment/solution to which the metal is exposed. The OCP gives the nobility of the endodontic file and reflects its ability to be oxidized/corroded with greater values indicating less likelihood to be oxidized. The Icorr is a measure of the corrosion rate of the endodontic file in that solution and is reflective of the amount of metal ions being released (dissolution), with a greater Icorr regarded as detrimental. The null hypotheses were that there would be no difference in the electrochemical behavior of the tested files, within and between solutions.

MATERIAL AND METHODS

Ten different NiTi endodontic file brands were tested: Twisted Files, K3 and K3XF (Kerr Endodontics, Orange, CA), ProFile, ProFile Vortex, and Vortex Blue (Dentsply Tulsa Dental Specialties, Tulsa, OK), HyFlex CM and HyFlex EDM (Coltene/Whaledent, Cuyahoga Falls, OH), and EndoSequence and EndoSequence CM (Brasseler USA, Savannah, GA) ($n = 10$/brand/solution). All files were of size 40 with a 0.04 taper. The 12 mm closest to the tip of each file was isolated for testing via nail polish covering the reminder of the file shaft. Each file was subjected to electrochemical testing in 5.25% NaOCl and saline (0.9% NaCl) at room temperature (22 ± 1°C) via a potentiostat (Interface 1010E; Gamry Instruments, Warminster, PA) coupled to a computer with associated software (Gamry Instruments). The electrochemical tests were completed using 3 electrodes with a saturated calomel electrode as the reference electrode, a graphite rod as the counter electrode, and each individual file as the working electrode. For each, the OCP was observed for 60 minutes, followed by a cyclic polarization test from −300 to 700 mV (vs OCP) and back to −300 mV at a scan rate of 1 mV/s. The thermodynamic and kinetic electrochemical behavior of the file was evaluated by 2 parameters, the OCP at 1 hour and Icorr, respectively. In addition to the quantitative measures, the cyclic polarization curves were also evaluated for the presence of a pitting corrosion loop. Due to unequal variances (Levene's test; $P < .01$), the OCP and Icorr were compared using the nonparametric Kruskal-Wallis test with pairwise comparisons via the Wilcoxon rank-sum test ($P < .05$) using statistical software (R version 4.2.2; The R Foundation). Spearman's correlations between measures were also computed.

RESULTS

The OCP at 1 hour and the Icorr in both solutions are shown in Table 1. Significant ($P < .05$) differences were observed between some brands for all 4 measures. ProFile Vortex and EndoSequence were the most noble (positive potentials) in saline, and EndoSequence and Vortex Blue were the most noble in NaOCl. Conversely, Twisted Files were the most active/anodic (most negative potentials) in both solutions. All file brands except for ProFile exhibited significantly ($P < .05$) greater OCPs in NaOCl compared to saline. Overall Icorrs in saline were very small and consistent, ranging from an average of 1.1 nA for Twisted Files to 15.2 nA for ProFile. Reflecting a more aggressive environment, much greater Icorrs were observed in NaOCl compared to saline; this was significant ($P < .05$) for all files except HyFlex EDM. A weak correlation between OCP and Icorr was observed in saline ($r_s = 0.21$, $P = .04$) and NaOCl ($r_s = 0.36$, $P = .0002$). Additionally, a moderate correlation ($r_s = 0.43$, $P < .0001$) between OCP in saline and NaOCl and a weak correlation ($r_s = 0.36$, $P = .0002$) between Icorr in saline and NaOCl were found.

Figure 1 displays representative OCP versus time graphs for all 10 file brands in both solutions. Potentials plateaued to a relatively constant value within 20 minutes of exposure for most files. Figure 2 shows multiple representative cyclic polarization plots. The cyclic polarization graph (Fig. 2*A*) has forward (toward more positive potentials) and reverse (toward more negative potentials) scans. It begins with cathodic currents, followed by transitioning to anodic currents at the corrosion potential, which is analogous to the OCP. With further increases in potential, current increases although for alloys that form passive layers, the current may remain relatively unchanged/vertical until the alloy reaches a potential whereupon the passive layer breaks down, if it does. When the potential reaches +700 mV versus OCP, the reverse scan begins. If this anodic current is greater than that of the forward scan, a pitting

TABLE 1 - OCP at 1 Hour, Icorr, and Percent of Specimens With Pitting Loops (%) in Saline and 5.25% NaOCl

	OCP (mV vs SCE) [mean (SD)]		Icorr (nA) [mean (SD)]		Pitting (%) ($n = 10$)	
File	Saline	NaOCl	Saline	NaOCl	Saline	NaOCl
Twisted Files	−219 (24) E	−19 (41) F	1.1 (0.4) A	41.2 (13.0) B	10	0
K3	−47 (23) C	181 (36) D	2.8 (1.2) C	100.1 (29.2) E	70	0
K3XF	−104 (30) D	10 (55) F	1.7 (0.5) B	39.5 (22.0) B	40	0
ProFile	−14 (97) AB	44 (27) E	15.2 (16.6) DE	59.4 (9.0) CD	60	0
ProFile Vortex	60 (73) A	425 (181) B	14.0 (25.2) DE	646.9 (402.7) G	10	0
Vortex Blue	−100 (63) CD	481 (114) A	6.8 (7.2) D	434.9 (112.8) G	100	0
HyFlex CM	−88 (66) CD	298 (249) BCDE	3.0 (2.4) BC	180.8 (259.6) ABDEF	100	0
HyFlex EDM	−111 (48) D	419 (64) C	13.4 (12.6) E	18.6 (12.8) A	100	0
EndoSequence	37 (37) A	516 (5) A	7.2 (3.0) DE	220.0 (130.4) F	90	0
EndoSequence CM	−1 (35) B	412 (168) ABC	1.2 (0.7) A	40.9 (39.6) ABC	50	0

Icorr, corrosion current; NaOCl, sodium hypochlorite; OCP, open circuit potential.
For each column, different letters indicate statistical significance ($P < .05$). Letters earlier in the alphabet signify a more favorable measure (eg, an "A" indicates a more positive OCP and an "A" in Icorr denotes the lowest corrosion current). OCP units are millivolts measured versus the saturated calomel electrode or reference electrode. Corrosion current units are in nanoAmperes.

loop is formed and localized corrosion occurs. Otherwise, the current remains anodic until the zero current potential is reached and current reverts back to cathodic. A pitting loop was only observed in saline solution and its frequency varied by brand of file (Table 1). Examples of pitting loops are observed for Vortex Blue in Figure 2*D*, HyFlex CM and HyFlex EDM in Figure 2*F*, and EndoSequence in Figure 2*H*.

DISCUSSION

The null hypotheses that there would be no difference in the electrochemical behavior of the NiTi files, within and between solutions of normal saline and 5.25% NaOCl, were rejected. Significant differences in OCP and Icorr were observed among the 10 files tested and the behavior, corrosion values, and rankings of the files in the 2 solutions differed. A dearth of information is published on the electrochemical behavior of NiTi endodontic files, so comparison to other studies is limited. Aside from Costa et al[36], other studies evaluating NiTi files via potentiodynamic testing were limited to 1 brand or experimental files[34,35], unlike the present study that evaluated 10 commercial files. Costa et al evaluated 2 heat-treated and 2 nonheat-treated NiTi files and found differences between the files but no clear distinction between the 2 groups. Similarly, files heat-treated after manufacturing (eg, HyFlex CM, Vortex Blue, EndoSequence CM) were generally not electrochemically distinguishable as a group from non-heat-treated files (eg, K3, ProFile, EndoSequence) in the present research. Primarily due to titanium, NiTi alloy will spontaneously form a passive oxide surface layer, something all files will possess, that helps protect it from corrosion. However, manufacturing processes may alter the thickness and quality of the passive layer. Depending on heat-treatment parameters, the oxide layer will usually be thicker in heat-treated NiTi, but a thicker oxide layer may not always result in greater corrosion resistance or less ion release because the quality of the oxide layers can be compromised and possess more defects[40]. Files with surface alterations (eg, EndoSequence with electropolishing and HyFlex EDM with a hardened surface via spark erosion[41]) also did not consistently display more favorable electrochemical behavior as compared to manufacturer counterpart files (EndoSequence CM and HyFlex CM). While electropolishing may produce a uniform, smooth and homogenous oxide layer[42], it did not increase EndoSequence's corrosion resistance as judged by the Icorr observed in either solution and pitting susceptibility in saline.

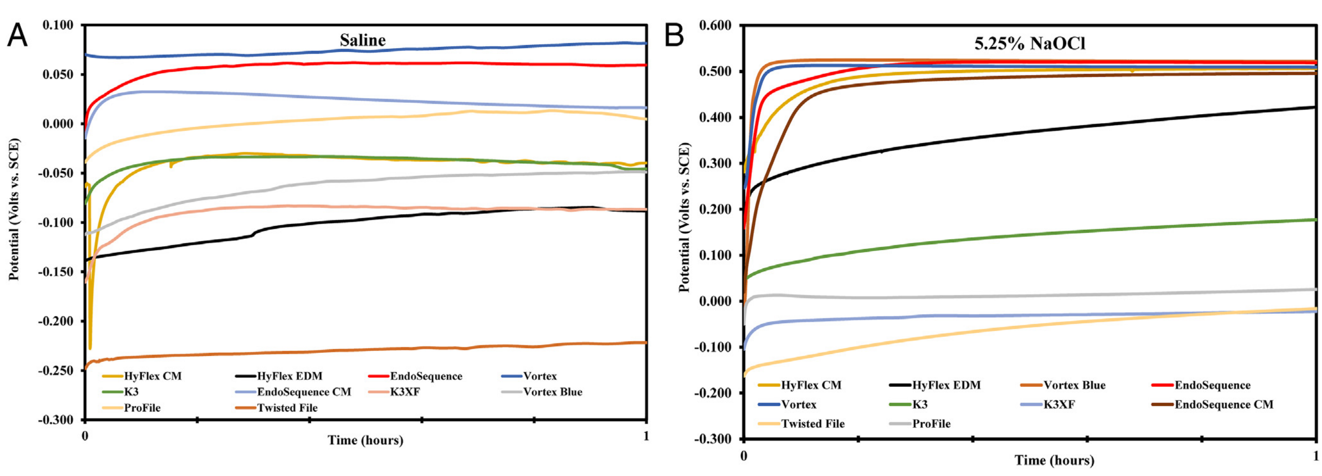

FIGURE 1 – Representative Open Circuit Potential versus Time curves in (*A*) saline and (*B*) 5.25% NaOCl.

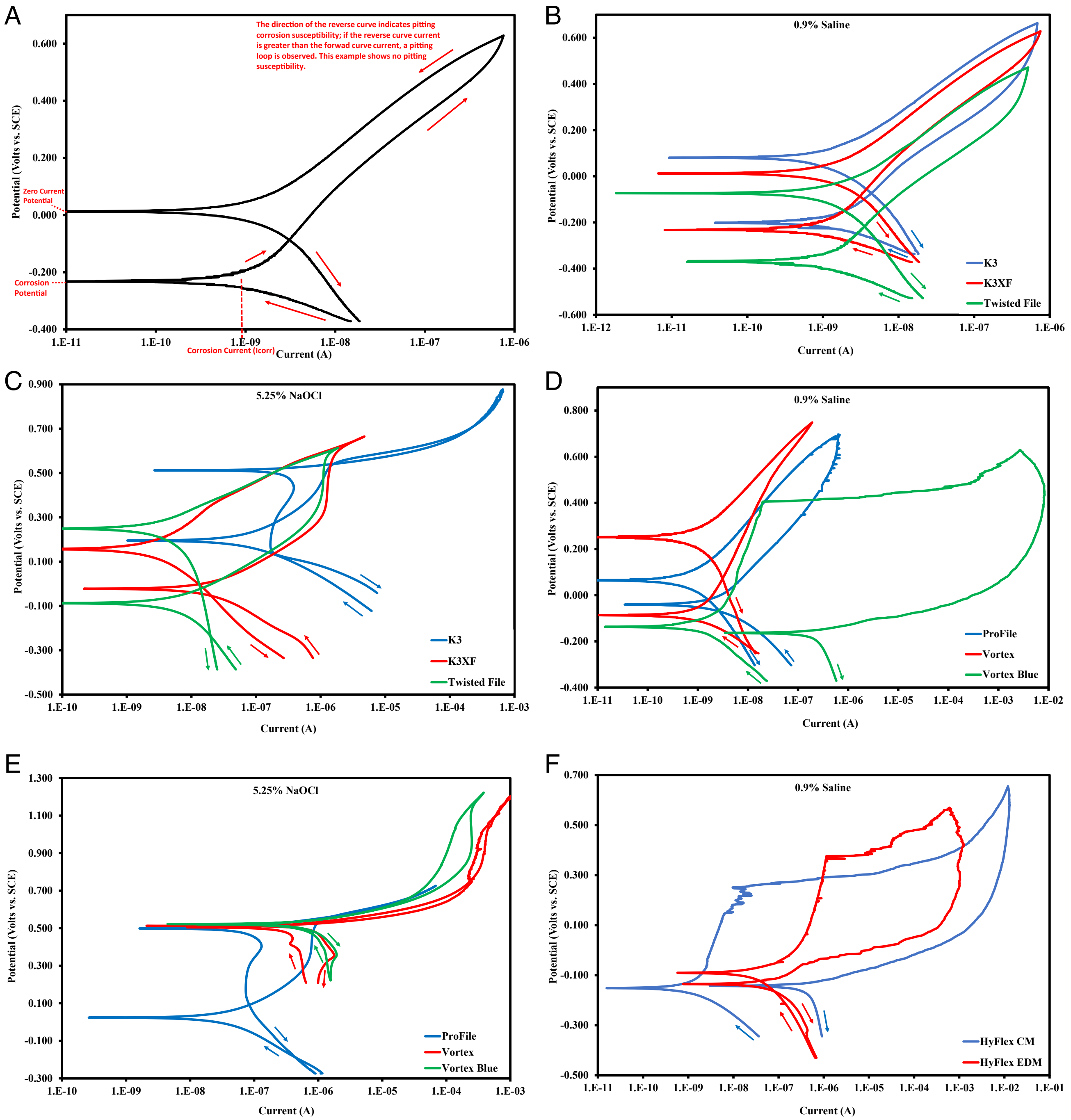

FIGURE 2 – Representative cyclic polarization curves. (*A*) Annotated typical curve, (*B*) Kerr files in saline, (*C*) Kerr files in 5.25% NaOCl, (*D*) Dentsply files in saline, (*E*) Dentsply files in 5.25% NaOCl, (*F*) Coltene files in saline, (*G*) Coltene files in 5.25% NaOCl, (*H*) Brasseler files in saline, and (*I*) Brasseler files in 5.25% NaOCl. NaOCl, sodium hypochlorite.

Of the 2 electrochemical parameters tabulated, Icorr may be of greater significance as it relates to the actual corrosion rate of the file whereas OCP indicates a tendency to corrode. The corrosion rate indicates the amount of corrosion and is directly proportional to the amount of metal ions being released. Only a weak correlation between Icorr in saline and NaOCl was found, meaning that some files were consistent in their relative corrosion resistance in both solutions, but this was not true for all files. Specifically, Kerr's Twisted Files was in the lowest statistical ranking in saline Icorr and in the second lowest statistical ranking in NaOCl, while Kerr's K3XF was in the second lowest Icorr ranking in both solutions, suggesting that Kerr's "R-phase technology" heat-treatment may influence corrosion rates. Conversely, the 3 Dentsply files tended toward greater corrosion rates, possibly implicating the raw NiTi stock material having an influence.

Endodontic files during clinical use for treatment of curved canals are subject to low cycle fatigue and may not be exposed to NaOCl for long periods of time. Nevertheless, categorically, fatigue resistance decreases in

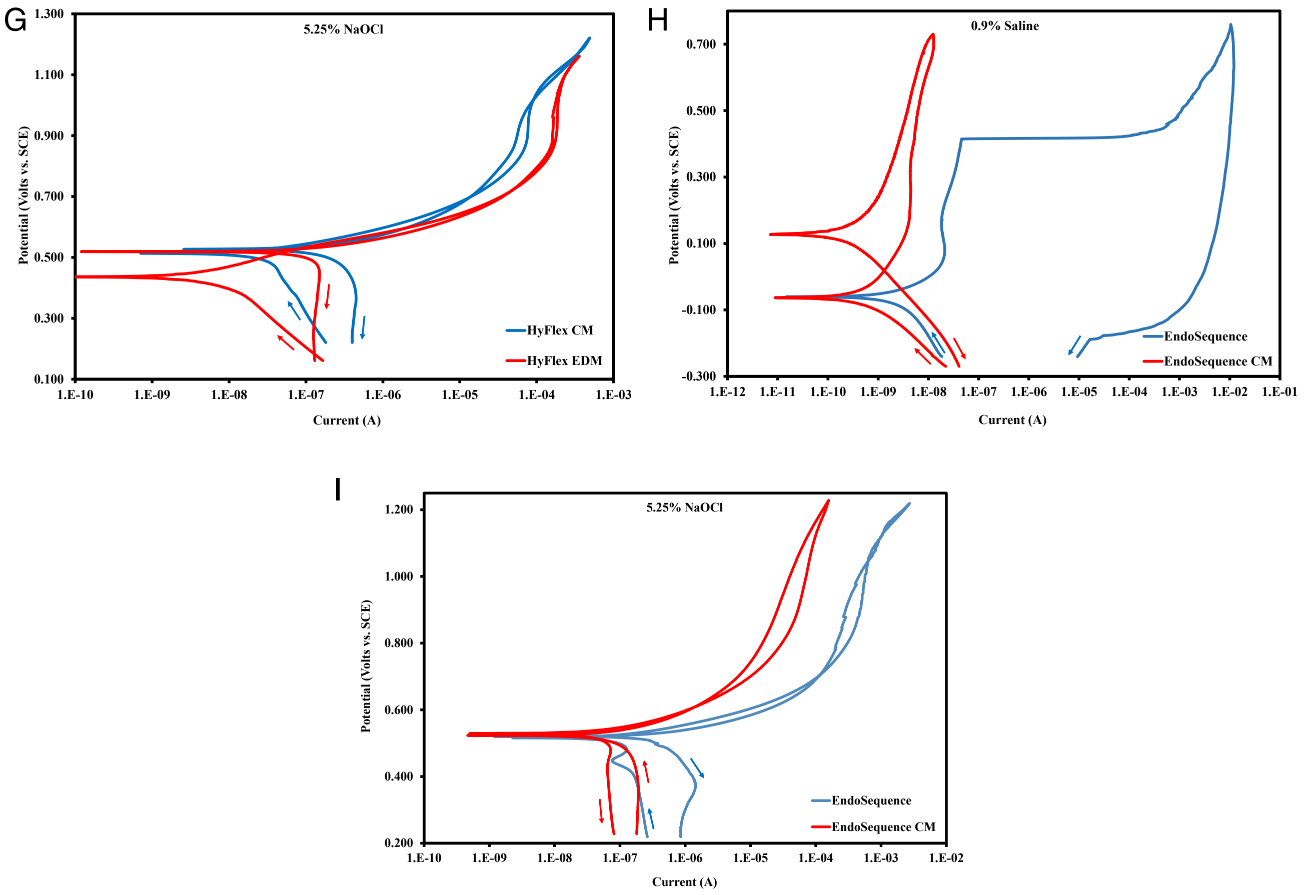

FIGURE 2 – Continued

more aggressive environments[43] and many studies, but not all, show a decrease in file fatigue life when exposed to NaOCl[22,24,25,27–33]. Relating the present electrochemical results to published cyclic fatigue studies is problematic due to the large methodological variance in the fatigue studies, as well as NaOCl exposure conditions. Nevertheless, a low corrosion rate in NaOCl would be preferred to limit the deleterious effect of NaOCl exposure on fatigue resistance. Similarly, for separated files unable to be retrieved from the tooth, exposure to biological fluids may cause corrosion of the file and release of ions. These ions have the potential to promote inflammation[44] and be toxic to cells[39]. For the 10 files evaluated in the present study, low average Icorrs (1–15 nA) were measured in saline, alleviating some concern that corrosion of fractured, nonretrievable NiTi file segments would negatively impact endodontic outcome. This is congruent with Spili et al[45] that found endodontic outcome prognosis was not significantly affected by the presence of a retained fractured instrument when managed by skilled endodontists.

The potentiodynamic cyclic polarization curves displayed pitting loop phenomena in the files exposed to saline, but not NaOCl which is consistent with 2 other studies. Bonaccorso et al[35] observed pitting in nonsurface treated and electropolished NiTi files in 0.9% NaCl solution, whereas Darabara et al[34] found no pitting was present for a NiTi file when tested with cyclic polarization in 5.25% NaOCl. Though the NaOCl solution may be considered more aggressive, as witnessed by the greater Icorrs when compared to saline, its high pH may have contributed to a more stable passive layer[46] that was less likely to be disturbed on a local level. It should also be pointed out that some of the observations of pitting in endodontic files when exposed to NaOCl may be influenced by the duration of exposure[20,23], sometimes with clinically unrealistic amounts. Though pitting may be more likely to occur with poor surface conditions (ie, those with defects), the electropolished EndoSequence files were still susceptible to pitting in saline, consistent with previous research[35].

Observation of the data in Table 1 shows the OCP and Icorr measurements sometimes exhibited a relatively high standard deviation for some brands, especially in the NaOCl solution. Reasons for this may include: Electrochemical data naturally have some scatter; no data were discarded, thus sometimes only 1 or 2 files may have produced the larger variance, and endodontic files are known to have heterogeneity in surface condition[6,47–49]. Further, it should be noted that Icorr is reported in nA and not reported as the more typical corrosion current density (nA/cm^2) where the exposed surface area is used to normalize the measured current. In general, a greater surface area will produce a greater current. Given the difficulty and imprecision of measuring the surface area of the unique file designs, instead, a standard length (12 mm) of file was chosen to allow better comparison between files. Another limitation of the present study is that the conjoint influence of stress and corrosion were not measured. Stress typically increases corrosion rates[50] and endodontic files would experience stress when instrumenting curved canals. Furthermore, OCP was measured for 1 hour and then the potentiodynamic scan was conducted to allow for corrosion cell stability and measurement accuracy, typical for this type of electrochemical testing. A file's exposure to NaOCl in the canal will be for a much shorter period of time whereupon the characteristics of the NaOCl are also subject to change[51]. Conversely, a file entombed within a canal may be exposed to biological fluid for long or indefinite periods of time. Furthermore, saline was used in this study to mimic this environment because chlorine is ubiquitous and one of the most corrosive agents in biological fluids;[52] however, the fluid an entombed file would be exposed to would be far more complex. How these aspects influence corrosion clinically remain to be evaluated.

CONCLUSIONS

Under the conditions of this study, significant differences in electrochemical properties were observed among the 10 brands of NiTi files. Overall, the files exhibited low corrosion rates in saline with greater corrosion rates in NaOCl. There was not a clear trend between conventional, heat-treated, or surface-treated files with respect to electrochemical properties.

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The authors deny any conflicts of interest related to this study.

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