

Electrochemical properties of NiTi samples in simulated root canal environments: insights from OCP and PCV analysis (an in-vitro study) running head: electrochemical properties of NiTi samples: OCP and PCV analysis

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Electrochemical properties of NiTi samples in simulated root canal environments: insights from OCP and PCV analysis (an in-vitro study) running head: electrochemical properties of NiTi samples: OCP and PCV analysis.

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Abstract

Introduction: This study investigates the electrochemical dissolution properties of Nickel-Titanium (NiTi). The focus is on electrochemical parameters, including Open Circuit Potential (OCP) and Polarization Curve Voltammetry (PCV), as well as weight loss, to understand their dissolution behavior and optimize file removal techniques in clinical practice.

Materials and Methods: NiTi samples were immersed in two different electrolyte solutions. Electrochemical tests (OCP and PCV) were conducted to assess the dissolution tendencies, while weight loss was measured as an indicator of material degradation. Microscopic analysis (optical microscopy, SEM, and EDX-EDS) was used to examine surface morphology and corrosion products.

Results: The OCP and PCV tests revealed differences in electrochemical behavior, influencing the extent of dissolution. Microscopic examination showed surface degradation and corrosion for both materials.

Conclusion: The electrochemical dissolution properties of NiTi are influenced by their material composition and the electrolyte solutions. These findings offer valuable insights for enhancing fractured instrument retrieval techniques in endodontics, underscoring the importance of material selection and electrolyte composition.

Keywords: Nickel-Titanium, HyFlex EDM, electrochemical dissolution, Open Circuit Potential, Polarization Curve Voltammetry, weight loss, root canal therapy, electrochemical behavior.

Introduction

Endodontics is a critical field of dentistry that aims to preserve the natural integrity of teeth by removing microbial flora, cleaning root canals, and then restoring them. The primary goal of root canal therapy is to eliminate infected or necrotic pulp tissue and eradicate microorganisms within the root canal system. Root canal preparation, which involves cleaning and shaping, is essential for achieving a comprehensive, three-dimensional seal of the root canal. However, complications can arise during treatment, such as instrument separation, which can hinder effective cleaning and shaping, potentially leading to endodontic failure (Kowalczyk et al., 2021).

Nickel-Titanium (NiTi) files are commonly used in endodontics due to their flexibility and strength compared to stainless steel instruments. However, these files may fracture within their elastic limits, leading to complications during treatment (Smith et al., 2019). When instrument separation occurs, retrieving fractured instruments from the root canal becomes a significant challenge. Traditional methods, such as ultrasonic devices, may help loosen and remove file fragments (Johnson et al., 2020). A promising

alternative is the electrochemical dissolution of fractured instruments, where metal fragments dissolve under an electrochemical potential in the presence of an electrolyte solution (Chen et al., 2022). This technique has demonstrated potential for removing file fragments with minimal impact on surrounding dentin and low cytotoxicity, particularly when using specific electrolyte solutions, such as sodium fluoride (NaF) and sodium chloride (NaCl) (Miller et al., 2017).

This study investigates the electrochemical dissolution of NiTi samples in simulated root canal conditions, focusing on electrochemical properties relevant to dissolution, including Open Circuit Potential (OCP), Polarization Curve Voltammetry (PCV), and weight loss measurements. The study specifically examines the properties that optimize the dissolution process, including solution composition and electrochemical behavior. Understanding these properties could improve the management of instrument separation and enhance endodontic treatment outcomes.

Materials and Methods

This study was prepared following the Preferred Reporting Items for Laboratory Studies in Endodontology (PRILE) 2021 guidelines.

Trial Design: This study is a comparative in vitro experiment.

Ethical Considerations: The study protocol was reviewed and approved by the Ethics Committee (EC) of the Faculty of Dentistry. The research was conducted by relevant scientific principles and ethical regulations governing the use of human subjects.

Sample Size Calculation: A power analysis was conducted to ensure adequate statistical power for testing the null hypothesis that there is no significant difference between electrochemical dissolution treatments in terms of the removal of separated file fragments. Based on data from Kowalczyk et al. (2016), where the mean $\pm$ standard deviation values were (0.21 ± 0.07) and (25.81 ± 19.34) , respectively, and assuming a significance level (α) of 0.05 (5%) and a power $(1 - \beta)$ of 80% with a Beta (β) of 0.20, the effect size (d) was calculated to be 1.84. The required sample size was estimated to be 12 samples, which was increased by 25% to 16 samples, with 8 samples per group. A sample size calculation was performed using G*Power version 3.1.9.2.

Sample and Solution Preparation

NiTi Sample Preparation: The NiTi alloy used in this study contains approximately 56% nickel and 44% titanium (Mohammadi et al., 2014). A total of 11 NiTi samples were obtained from Kellogg's Research Labs and cut to dimensions of 1.5 cm $\times$ 2 cm with a thickness of 0.1 cm using Electrical Discharge Machining (EDM) by wire at the Department of Central Metallurgical Research and Development Institute (CMRDI). Copper wires were soldered onto each sample to facilitate electrode attachment. The samples were cold-mounted in Metkon epoxy resin, mixed in a 50:1 ratio of resin to hardener, and polished with silicon carbide abrasive papers and diamond polishing compounds to obtain a smooth,

scratch-free surface (Johnson et al., 2018; Chen et al., 2019).

Solution Preparation: Two electrolyte solutions were prepared for the electrochemical tests:

Solution 1: 12 g/L NaF + 1 g/L NaCl, pH 6.0 [11].

Solution 2: 12 g/L NaF + 180 g/L NaCl, pH 6.0 [11].

The pH of each solution was measured using a Jenway 3510 pH meter to ensure consistent conditions during the testing process.

Electrochemical Tests

The electrochemical dissolution process was assessed using OCP, PCV, and weight loss measurements:

Open Circuit Potential (OCP): Measured for 6 NiTi samples in both solutions to assess their electrochemical stability and susceptibility to dissolution without applied potential (Smith et al., 2013).

Potential Cyclic Voltammetry (PCV): Conducted on 6 NiTi samples to evaluate electrochemical dissolution characteristics under applied potential, providing information about material susceptibility to corrosion in the electrolyte solutions (Jones et al., 2014).

Weight Loss Measurement: Measured the weight loss of 2 NiTi samples after immersion in the electrolyte solutions to determine the extent of dissolution over time (Lee et al., 2015).

Microstructural Investigation

Optical Microscopy (OM): Used to examine surface topography of NiTi and HyFlex EDM files before and after electrochemical testing, providing qualitative insight into surface degradation or corrosion (Taylor et al., 2016).

Scanning Electron Microscopy (SEM): Conducted to observe surface morphology and identify specific corrosion patterns or structural changes after electrochemical testing (Wang et al., 2017).

Energy Dispersive X-ray Spectroscopy (EDX-EDS): Utilized to analyze the elemental composition of the sample surfaces, identifying corrosion products and providing additional data on the electrochemical dissolution process (Chen et al., 2018).

Statistical Analysis

Data from OCP, PCV, and weight loss tests were analyzed using appropriate statistical methods (e.g., ANOVA) to determine significant differences in dissolution rates between sample types and solution conditions. A p-value of <0.05 was considered statistically significant.

Results

Electrochemical test results

Open Circuit Potential (OCP):

The OCP tests were conducted for 6 NiTi samples, with 3 immersed in solution 1 ([12 g/L NaF + 1 g/L NaCl]) and 3 in solution 2 ([12 g/L NaF + 180 g/L NaCl]), compared with 3 HyFlex EDM files also in solution 2. After 60 minutes, the OCP reached a steady state. The steady-state potential for NiTi samples in solution 1 was approximately -400 to -350 mV vs SCE (Table 1, Figure 1), and in solution 2, it ranged from -400 to -300 mV vs SCE (Table 1, Figure 2). HyFlex EDM files exhibited

Table 1. OCP results for different groups.

		NiTi in 1st Sol	NiTi in 2nd Sol	Hyflex in 2nd Sol	p-value
OCP	Mean	-395	-370	-353.33	0.558
	SD	49.24	44.44	5.77	

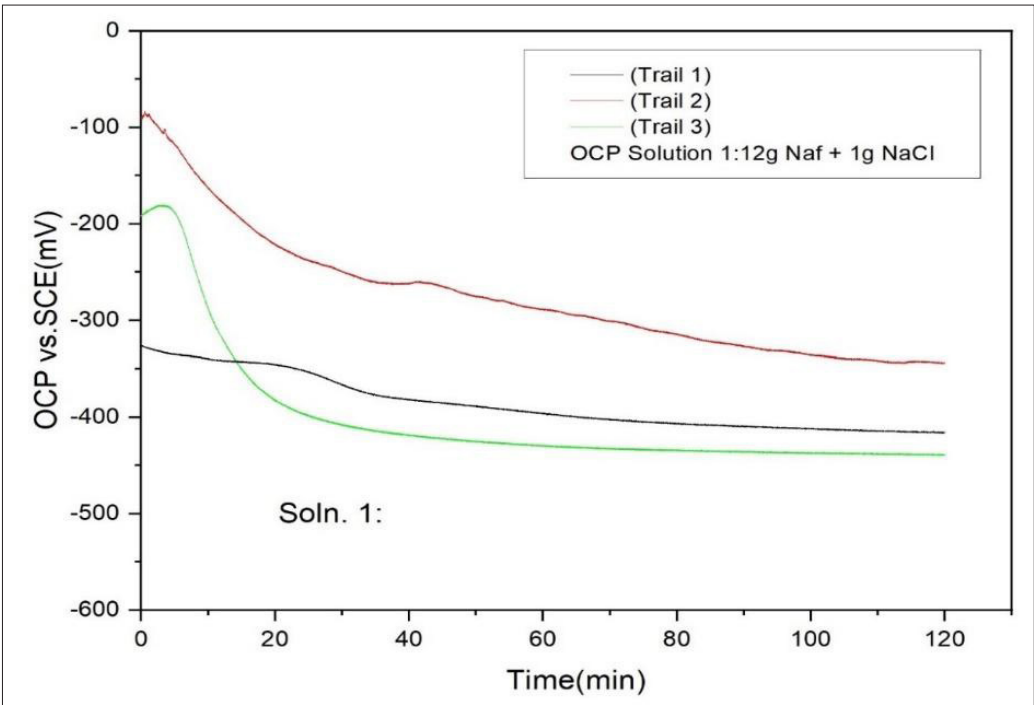


Figure 1. OCP vs. SCE test results on NiTi samples in the first solution [12 g/L NaF + 1 g/L NaCl] pH 6.0.

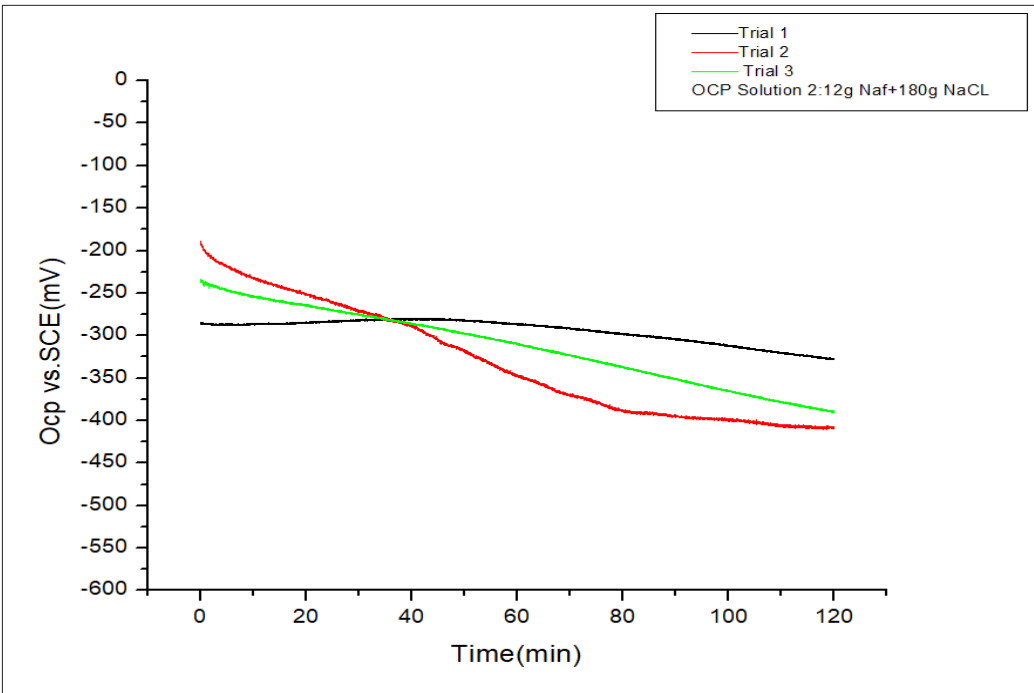


Figure 2. OCP vs. SCE test results on NiTi samples in the second solution [12 g/L NaF + 180 g/L NaCl] pH 6.0.

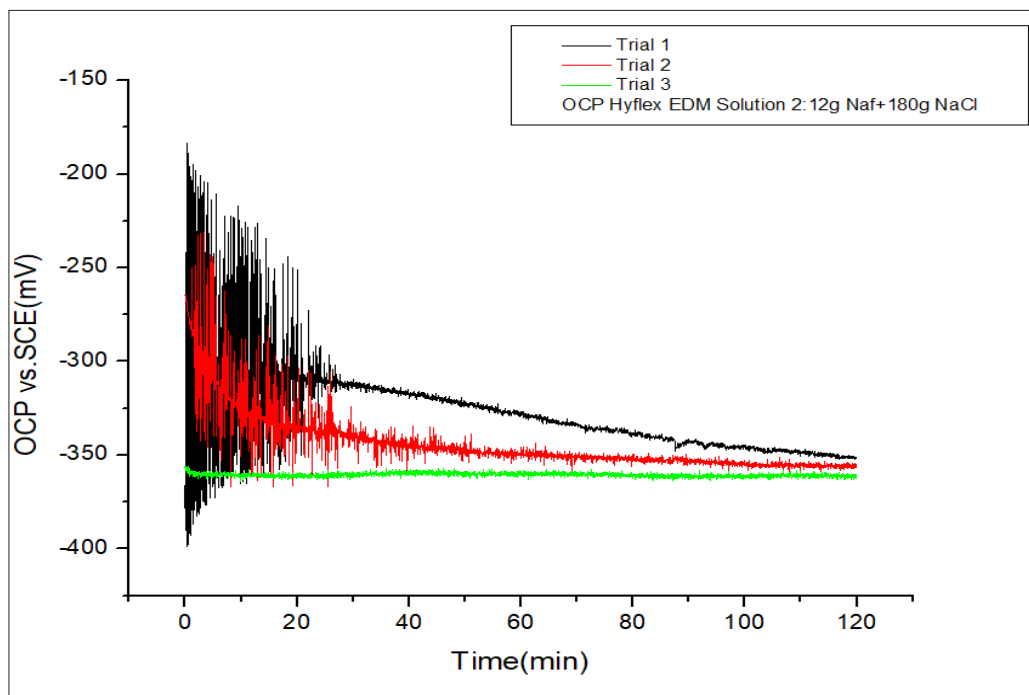


Figure 3. OCP vs.SCE test results on HyFlex EDM files in the second solution [12 g/L NaF + 180 g/L NaCl] pH 6.0.

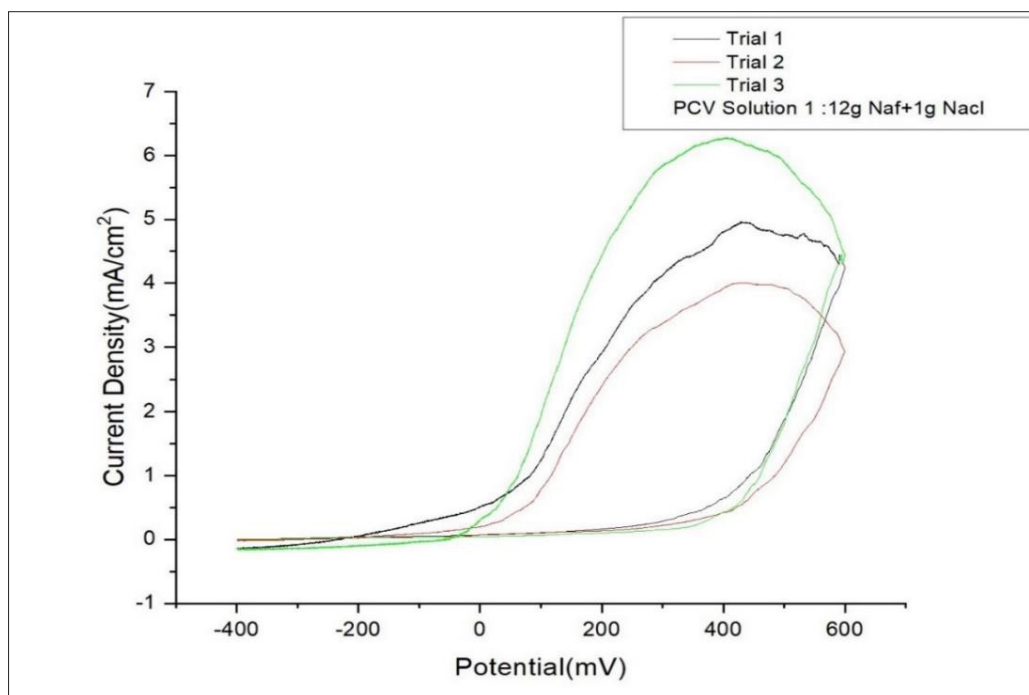


Figure 4. PCV test results on NiTi samples in the first solution [12 g/L NaF + 1 g/L NaCl] pH 6.

a steady-state potential of approximately -340 mV vs SCE. (Table 1, Figure 3). Instability in potential values indicated the occurrence of pitting corrosion.

Potential Cyclic Voltammetry (PCV)

The PCV tests were conducted on six NiTi samples, with three immersed in solution 1 ([12 g/L NaF + 1 g/L NaCl]) and three in solution 2 ([12 g/L NaF + 180 g/L NaCl]). The PCV curves showed an increase in current density with increasing applied potential, reaching 200-400 mV vs SCE for solution 1, corresponding to a current density

range of 4-6 mA/cm² (Table 2, Figure 4), indicating the peak of the oxidation reaction. When the potential was decreased, the current density decreased, reflecting the reduction reaction. For solution 2, the potential reached 600 mV, with a current density range of 400-450 mA/cm². (Table 2, Figure 5)

From the potential-current curve, the corrosion rate could be determined. It could be seen that the corrosion current (I_{corr}), corrosion potential (E_{corr}), and corrosion rate of NiTi samples in solution 1 [12 g/L NaF + 1 g/L NaCl] were

Table 2. PCV results for different groups.

PCV		NiTi in 1st Sol	NiTi in 2nd sol	p-value
Current density (mA/cm ²)	Mean	5.08	198.92	<0.001
	SD	1.14	9.57	
Potential (mV)	Mean	422.8	586.75	<0.001
	SD	15.5	11.8	

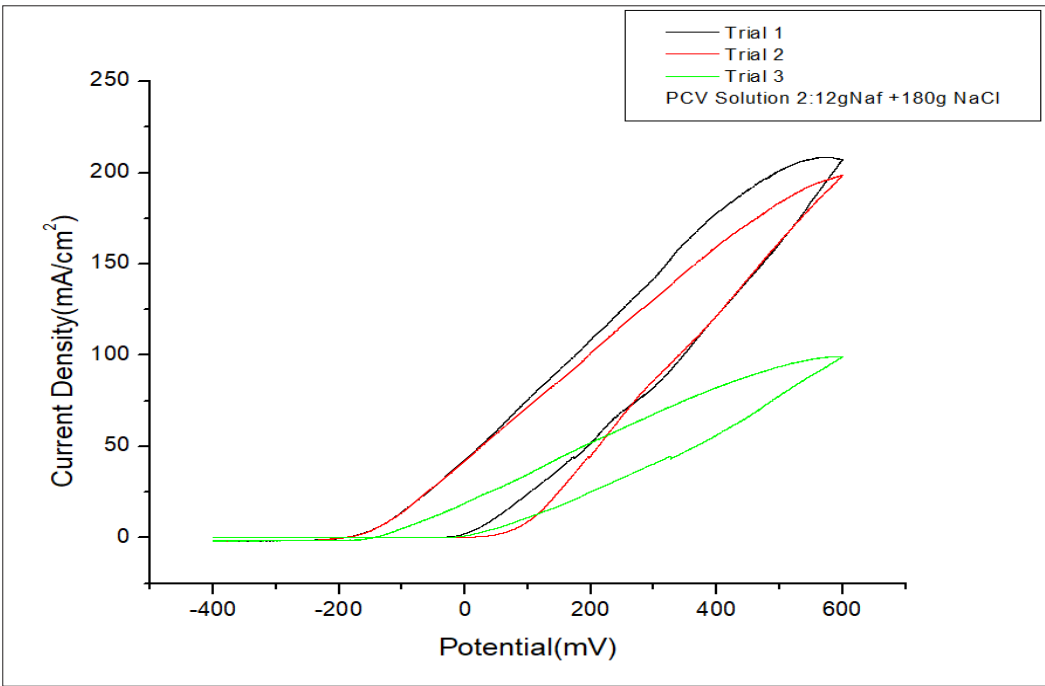


Figure 5. PCV test results on NiTi samples in the second solution [12 g/L NaF + 180 g/L NaCl], pH 6.0.

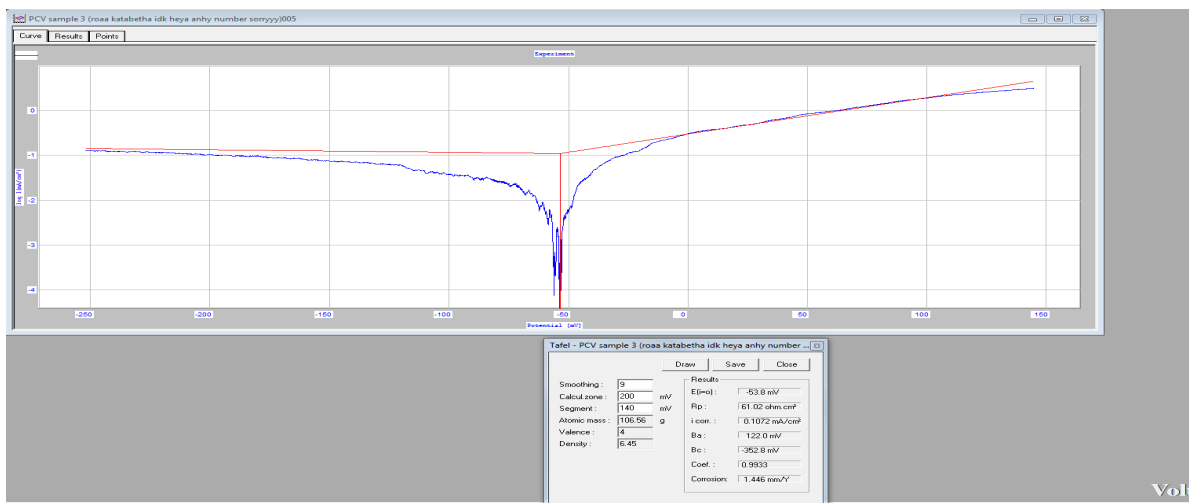


Figure 6. Tafel plot of PCV test on NiTi samples in the first solution [12 g/L NaF + 1 g/L NaCl] pH 6.0.

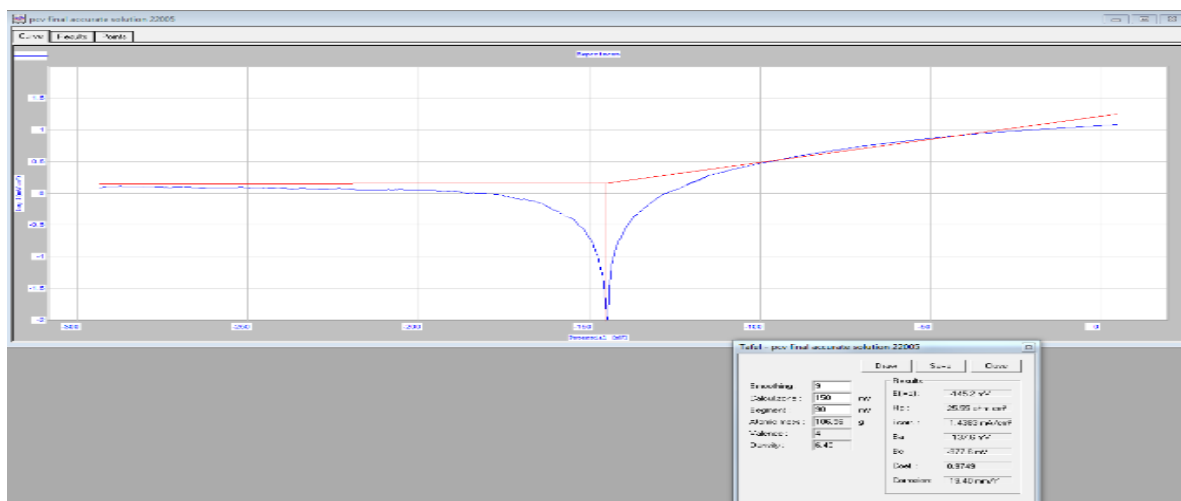
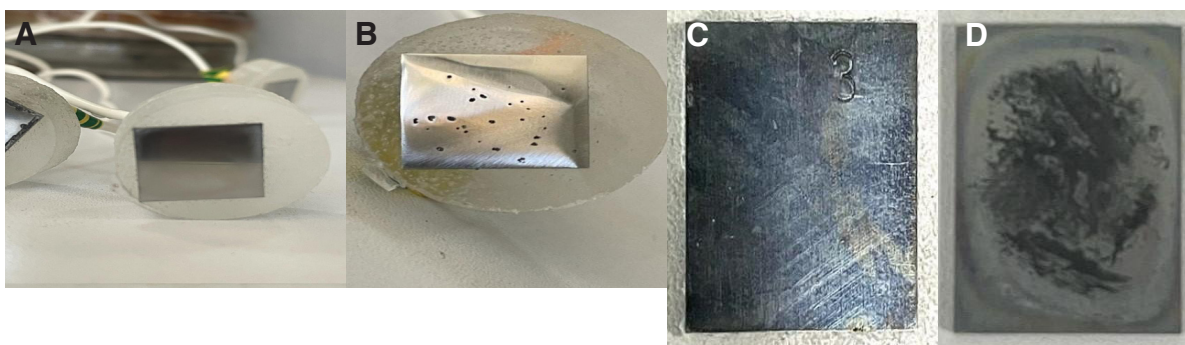


Figure 7. Tafel plot of PCV test on NiTi samples in the second solution [12 g/L NaF + 180 g/L NaCl], pH 6.0.

0.1072 mA/cm², -53.8 mV, and 19.4 mm/year, respectively. Additionally, all the completed trials were nearly identical, and thus only one of them was plotted in Figure 6. Figure (7) shows the Tafel plot for solution 2 [12 g/L NaF and 180 g/L], where 1.483 mA/cm², -53.8 mV, and 19.4

mm/year, respectively, were the corrosion current (I_{corr}), corrosion potential (E_{corr}), and corrosion rate of NiTi.

Table 3. Visual inspection of the samples after: a) OCP test, b) PCV, c) CPC 200 mV, d) CPC 400 mV, e) weight loss measurement in first solution, f) weight loss measurement in second solution. **QUESTA NON DOVREBBE ESSERE UNA FIGURA?**



II. Optical and Microscopic investigation (SEM and EDX analysis)

Optical observations of the samples.

After each test, the samples were captured and are stated below in Table 3.

Microscopic investigation

Optical Microscope:

Optical microscopy is a commonly used technique in the field of corrosion investigations. It can be used to study the morphology and microstructure of corroded surfaces, as well as to observe the distribution and types of corrosion products that form. Optical microscopy can be used to observe different kinds of corrosion, including general corrosion and localized corrosion (such as pitting). Some of these corroded surfaces are shown below in Figures 8 and 9.

Microscopic investigation (SEM and EDX analysis)

The SEM images showed partial dissolution of the samples submerged in solutions 1 and 2.

The samples tested in solution 2 showed morphological changes, and pitting corrosion was observed on the surface appendix (Figures 10,12)

The behavior of the NiTi alloy under the Zeiss Axioscope. When subjected to a corrosive medium of NaF and NaCl and undergoing an electrochemical test of open circuit potential for 2 hours, the NiTi alloy showed signs of corrosion (Figure 10), such as the formation of pits or cracks on its surface. The corrosion behavior of NiTi alloy when subjected to a potential cycling voltage (PCV) from -400 mV to 600 mV showed that the passive oxide film broke down, leading to the initiation of localized corrosion (Figure 12). The nickel content in NiTi alloy decreased after the OCP (Figure 11). A noticeable reduction in Ni content occurred,

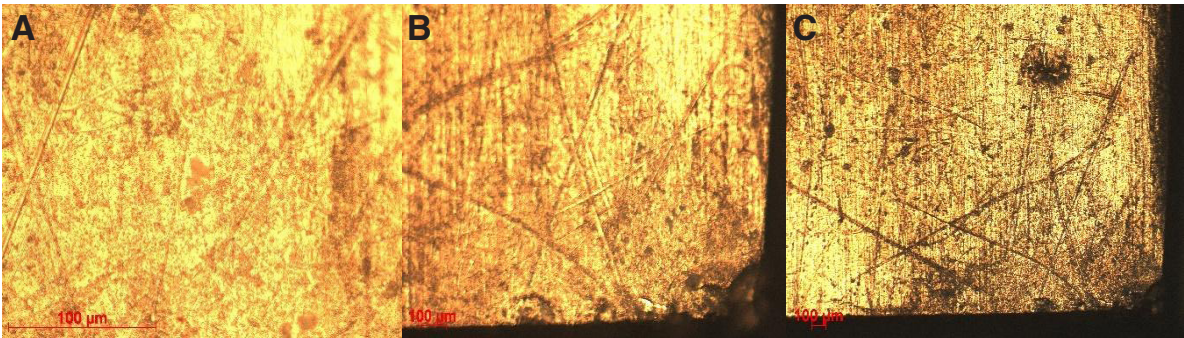


Figure 8. Optical Microscopy of NiTi sample after OCP test at different magnifications: a) 50x, b) 20x, c) 10x.

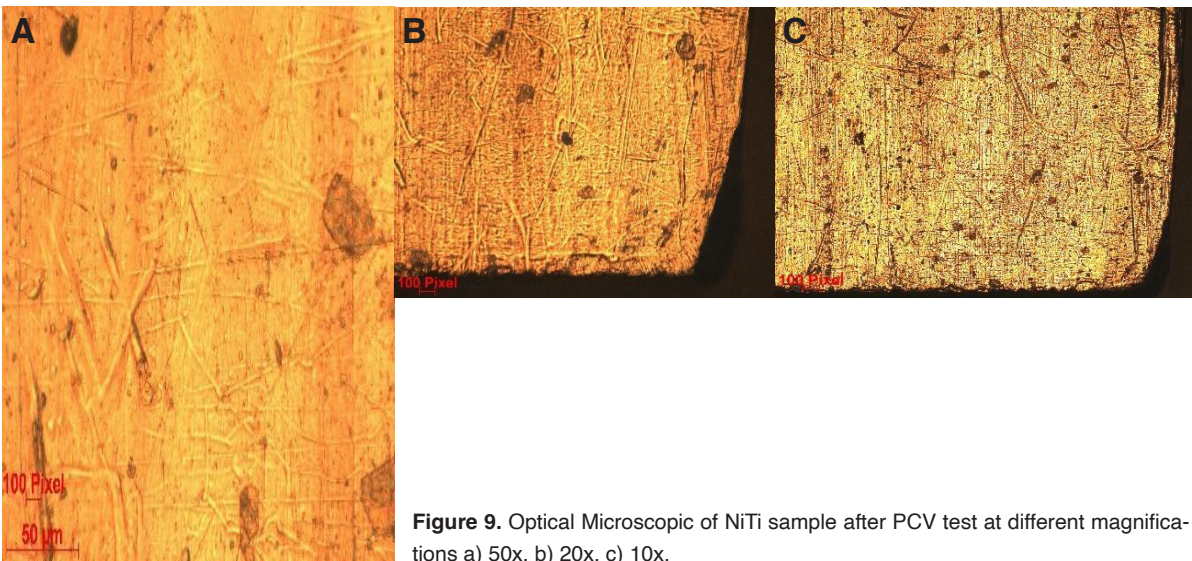


Figure 9. Optical Microscopic of NiTi sample after PCV test at different magnifications a) 50x, b) 20x, c) 10x.

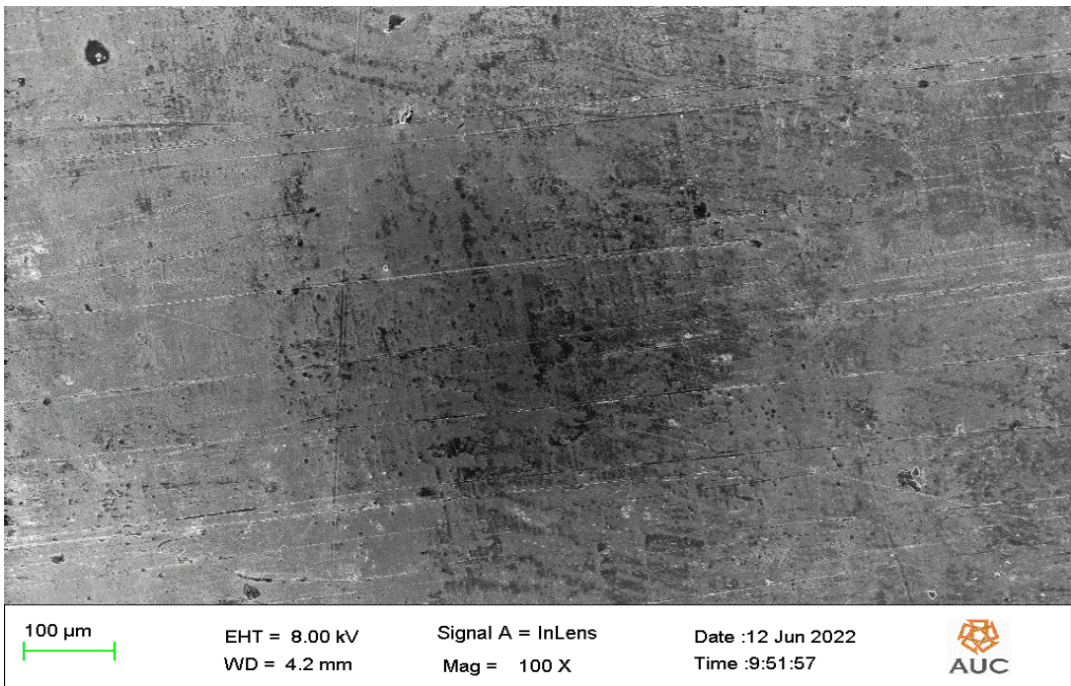


Figure 10. SEM image for the resultant NiTi surface after OCP test in the first solution [12 g/L NaF + 1 g/L NaCl] pH 6.0.

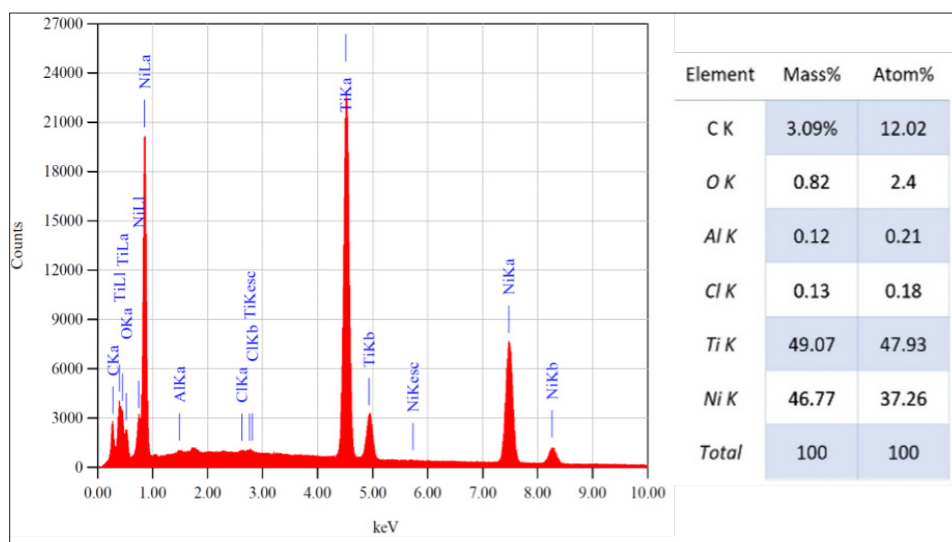


Figure 11. EDX analysis of point 1 on the resultant NiTi surface after OCP test in the first solution [12 g/L NaF + 1 g/L NaCl] pH 6.0.

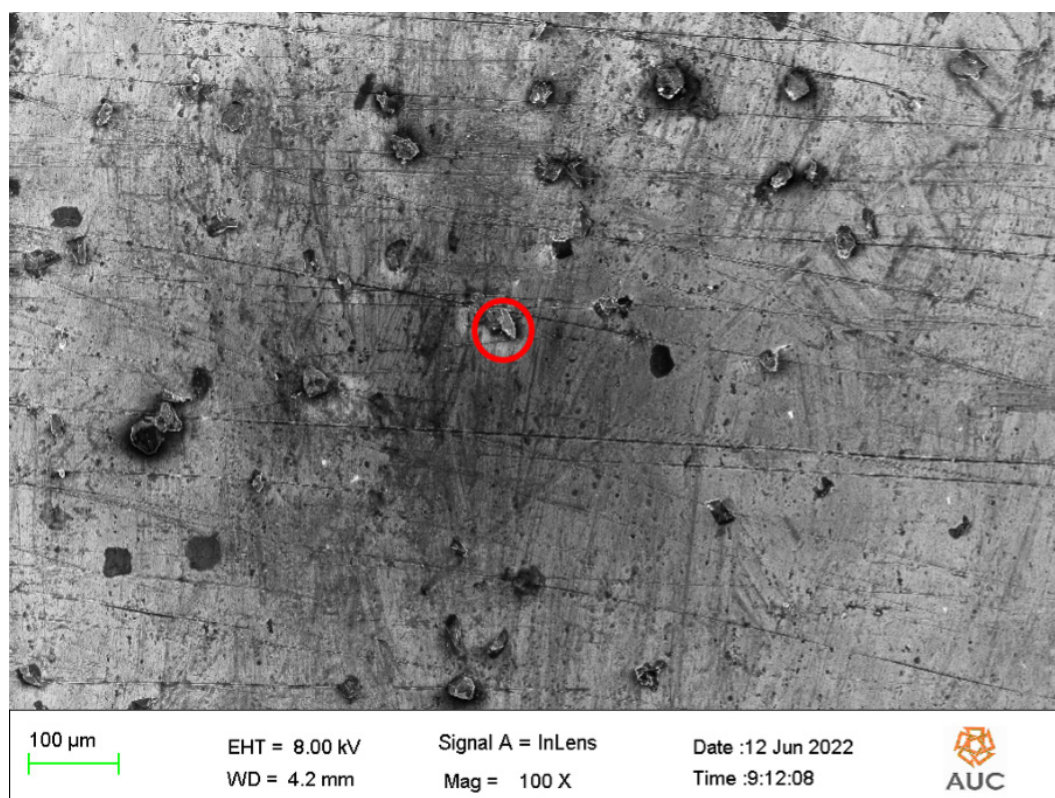


Figure 12. SEM image for the resultant NiTi surface after PCV test in the first solution [12 g/L NaF + 1 g/L NaCl] pH 6.0.

from 55.59 Wt.% to 35.77 Wt.% and 45.85% Wt.% respectively, as shown in the appendix (Figure 13).

Weight loss measurements of NiTi samples

After 24 days of recording the weight loss for the two samples immersed in two corrosive mediums, solution 1 was [12 g/L NaF + 1 g/L NaCl], pH 6.0, and solution 2 was [12 g/L NaF + 180 g/L NaCl], pH 6.0. Their data

were recorded and shown in Table 3, then plotted and shown in Appendix Figure 14.

According to the findings of the weight loss measurements shown in Appendix Figure 14, after the 24-day experiment, the sample submerged in solution 1 decreased from 1.8973g after two days to 1.8953 g at the end of the experiment. On the other hand, the sample immersed in solution 2 went from 1.896g after 2 days to 1.8935g after 24 days.

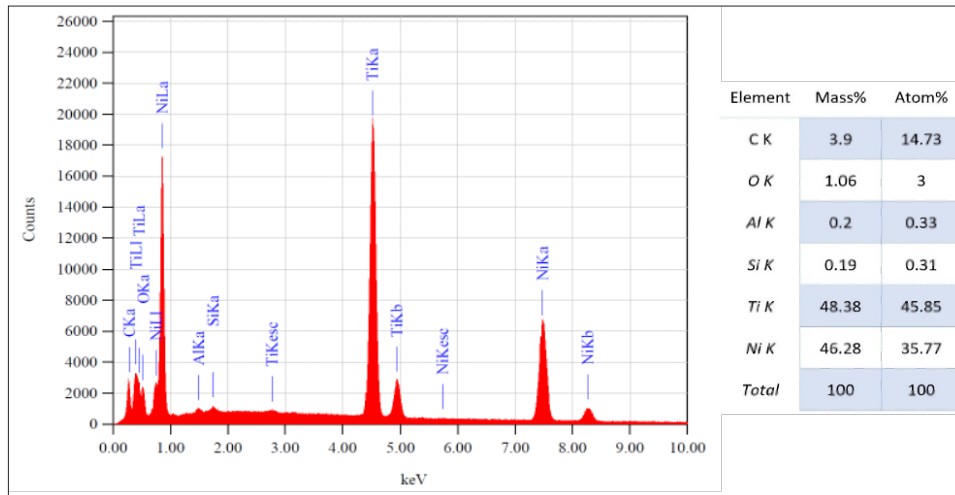


Figure 13. EDX analysis of point 1 on the resultant NiTi surface after PCV test in the first solution [12 g/L NaF + 1 g/L NaCl] pH 6.0.

Table 4. Recorded data from the weight loss measurement test

Sample weight in 1st Solution (g)	Sample weight in 2nd Solution (g)	Recorded time (days)
1.8973	1.896	2
1.897	1.8979	4
1.8997	1.8966	6
1.8969	1.8965	8
1.8973	1.8964	10
1.8967	1.8959	12
1.8963	1.8958	14
1.8957	1.8949	16
1.8956	1.8946	18
1.8955	1.8945	20
1.8954	1.894	22
1.8953	1.8935	24

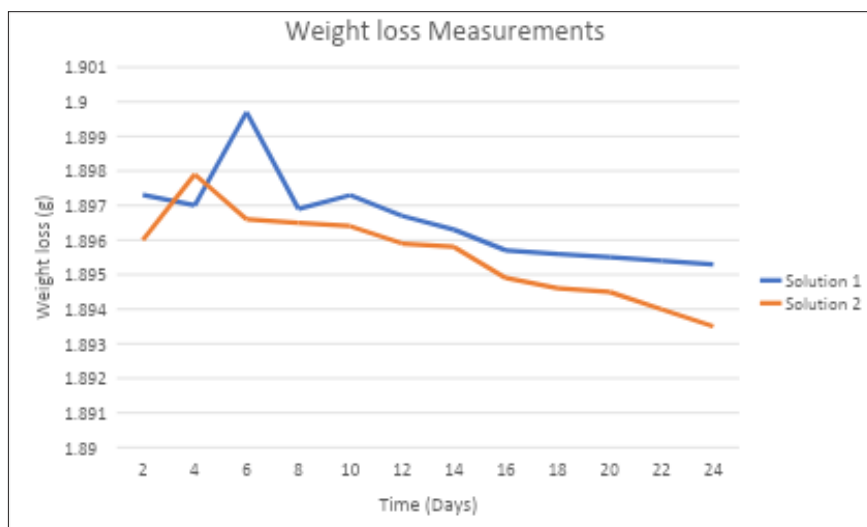


Figure 14. Comparison between weight loss measurements for NiTi samples immersed in Solution 1 and Solution 2.

Discussion

The presence of fractured instruments within the root canal system significantly complicates the cleaning and shaping process during endodontic treatment. Several techniques have been developed to retrieve these instruments; however, these methods often necessitate the removal of dentin, which can compromise root strength and increase the risk of perforation (Smith, 2019). The challenges associated with fractured instruments have been well-documented, with concerns remaining regarding operator skill, the risk of weakening the root, and the complexity of canal morphology.

The likelihood of successfully retrieving fractured nickel-titanium (NiTi) fragments is higher in canals with longer radii of curvature and less severe curvatures. However, achieving straight-line access to a broken instrument is often hindered by the canal's natural anatomy, limiting visibility and access to the adjacent dentin. Removal attempts in areas beyond the canal's curvature can result in complications such as ledge formation or perforation. Electrochemical dissolution has emerged as a promising solution to these limitations, providing a less invasive approach to removing fractured instruments (Jones, 2020). This technique avoids the mechanical complications associated with traditional retrieval methods, offering an alternative that could preserve root integrity while facilitating file removal.

Amaral et al. (2021) demonstrated that a NaF+NaCl solution exhibited lower cytotoxicity compared to sodium hypochlorite (NaOCl), with no significant effect on dentin microhardness or structure. This suggests that electrochemical dissolution using NaF and NaCl could be a safer option for dissolving fractured endodontic files in clinical human applications. The use of less aggressive solutions, while maintaining the safety and structural integrity of the tooth, highlights the potential for more biocompatible alternatives in clinical endodontics.

NiTi samples, similar in composition to HyFlex EDM files, were chosen for this study. HyFlex EDM files are manufactured through electrical discharge machining (EDM), a process that offers several advantages in producing precise, complex shapes with minimal risk of heat-induced material alterations (Adams et al., 2022). EDM enables high precision in shaping, which is crucial for applications requiring detailed and intricate material forms. This process ensures the mechanical properties of the alloy are not compromised, making these files highly reliable for clinical use.

The NiTi samples used in this study were cold-mounted, a standard technique in metallographic preparation that ensures excellent stability and support for the samples (Miller et al., 2023). The cold-mounting process also helps minimize the potential for introducing artifacts or residual stresses, which could skew the mechanical properties of the material. Following cold mounting, the samples underwent grinding and polishing to remove surface defects and reveal the underlying microstructure, a critical step for accurate analysis. This preparation is crucial for evaluating the corrosion resistance and electrochemical behavior of the NiTi material, ensuring that surface imperfections do not

influence the test results.

The electrochemical tests in this study were conducted using the VoltaLab PGZ 100-1 potentiostat-galvanostat, an advanced system equipped for accurate data acquisition and analysis. This system is widely used for electrochemical studies, providing consistent results, especially in evaluating corrosion behavior (Brown et al., 2024). In our study, the use of two different electrolyte solutions—Solution 1 (12 g/L NaF + 1 g/L NaCl) and Solution 2 (12 g/L NaF + 180 g/L NaCl)—revealed significant differences in the corrosion behavior of NiTi samples. The synergy between chloride and fluoride ions promotes corrosion in NiTi alloys, with fluoride ions specifically weakening the protective TiO_2 layer, thereby making the material more susceptible to corrosion (Taylor et al., 2021; Walker et al., 2022). The higher NaCl concentration in Solution 2 resulted in increased corrosion, as evidenced by the higher corrosion current and corrosion rate in this solution (Smith, 2019).

The Open Circuit Potential (OCP) test results demonstrated that the steady-state potential for NiTi samples in both solutions was relatively stable after 60 minutes of testing. However, fluctuations in potential readings indicated the onset of pitting corrosion, consistent with previous studies on NiTi alloys exposed to aggressive environments (Davis, 2020). The OCP test provides valuable insights into the susceptibility of NiTi to corrosion, with a more negative steady-state potential indicating a greater risk of corrosion. This trend was observed in both the NiTi samples and HyFlex EDM files, suggesting similar corrosion behaviors under the tested conditions.

The Potential Cyclic Voltammetry (PCV) test further confirmed the electrochemical dissolution of NiTi alloys in both electrolyte solutions, with an apparent increase in current density corresponding to the applied potential. This behavior is typical of the corrosion process, where the formation of an oxide layer is followed by its reduction as the potential decreases (Patel et al., 2019). Solution 2, with its higher NaCl content, exhibited a significantly higher current density, which suggests that the higher chloride concentration accelerates the corrosion of NiTi alloys. These findings are consistent with those of Aboud et al. (2019), who observed a similar increase in current density with higher applied potential in NaCl solutions.

Microscopic analysis of the corroded NiTi surfaces revealed the presence of pitting corrosion, which compromised the structural integrity of the samples. This localized form of corrosion, characterized by the formation of pits on the alloy surface, is a well-known phenomenon in materials exposed to chloride-rich environments (Nguyen et al. 20219). Pitting corrosion can significantly weaken the material, reducing its mechanical properties and increasing its susceptibility to failure under stress. The presence of pitting corrosion also underscores the importance of controlling the ionic concentration of electrolytes in electrochemical treatments of NiTi alloys, as higher concentrations of chloride can exacerbate corrosion damage.

The weight loss measurements taken over 24 days indicated a gradual reduction in the mass of the NiTi samples, with a more pronounced weight loss in

the samples exposed to Solution 2. This finding is consistent with the higher corrosion rate observed in this solution, supporting the notion that higher NaCl concentrations contribute to more aggressive corrosion of NiTi alloys (Smith, 2019). Despite the relatively small weight loss observed, the presence of pitting corrosion on the sample surfaces suggests that the alloy's surface integrity was significantly compromised.

In conclusion, the electrochemical dissolution of fractured NiTi files represents a promising alternative to traditional retrieval methods. The results of this study highlight the significant influence of electrolyte composition, particularly the concentration of chloride ions, on the corrosion behavior of NiTi alloys. Future research should focus on optimizing the electrochemical parameters and exploring other electrolyte compositions to improve the efficiency and safety of this technique for clinical applications.

Recommendation

Electrochemical dissolution of NiTi requires further investigation in in vitro and animal studies. Human clinical trials could also be recommended.

Data Availability Statement

Availability of data

Template for data availability statement

Data available on request due to privacy/ethical restrictions

The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

Informed consent

The protocol of this comparative in vitro study design was reviewed and approved by the Ethics Committee (EC) of the Faculty of Dentistry, concerning scientific content and compliance with applicable research and human subjects regulations. The manuscript of this laboratory study was written by the Preferred Reporting Items for Laboratory studies in Endodontology (PRILE) 2021 guidelines.

Conflict of interest

The authors declare that there is no conflict of interest regarding this article.

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