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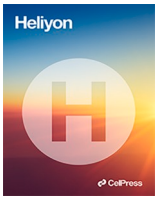

Research article

Microhardness and energy dispersive X-ray evaluation of dentinal walls following chemical and electrochemical dissolution of fractured nickel-titanium file: An in vitro study

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ABSTRACT

Purpose: Chemical dissolution of nickel-titanium (NiTi) files involves the application of a fluoride solution in direct contact with a damaged instrument, whereas electrochemical dissolution involves the application of an electrical current to the electrolyte, which accelerates fragment dissolution. This study aimed to determine the hardness and concentration of calcium and phosphorus (Ca and P) ions in dentinal walls following chemical and electrochemical dissolution of fractured ProTaper F2 files with a novel chemical solution.

Materials & Methods: Thirty human maxillary first molar palatal roots with fractured ProTaper F2 files in the middle third (length, 2.5 mm were divided into three groups according to the treatment techniques used with a novel solution (NaF 12 g/L + $NaCl_2$ 60 g/L + $MgCl_2$ 60 g/L + $CaCl_2$ 60 g/L) at pH 5: Group 1: distilled water (control group), Group 2: electrochemical dissolution, and Group 3: chemical dissolution using the novel solution. The novel solution was placed for 10 min using an electrochemical technique, and for 30 min in contact with the separated instrument in the chemical group. The Vickers microhardness test was performed in three areas: at 3, 6, and 9 mm from the apex, and an energy-dispersive X-ray test for both Ca and P ions was performed. The analysis of variance (ANOVA) and Tukey's tests were used for statistical analysis.

Results: According to the one-way ANOVA analysis, no difference was observed between the tested approaches ($P > 0.05$) in the three areas evaluated (3, 6, and 9 mm), with no difference in the Ca/P ratio between the tested groups.

Conclusion: Compared to the control group, the use of chemical and electrochemical dissolution methods with the novel solution did not affect dentin hardness or dentinal structure in terms of the Ca/P ratio, thereby indicating promising results while saving time.

1. Introduction

In the field of endodontics, the management of fractured nickel-titanium (NiTi) files inside a root canal can be challenging, especially in cases of persisting previous infection or inadequate cleaning and disinfection [1,2]. Prescribed protocols for the removal of a fractured file from the root canal may result in the development of complications and iatrogenic issues, such as root weakening and perforation [3–5]. Thus, research has focussed on conservative techniques for the removal of the fractured endodontic files, such as

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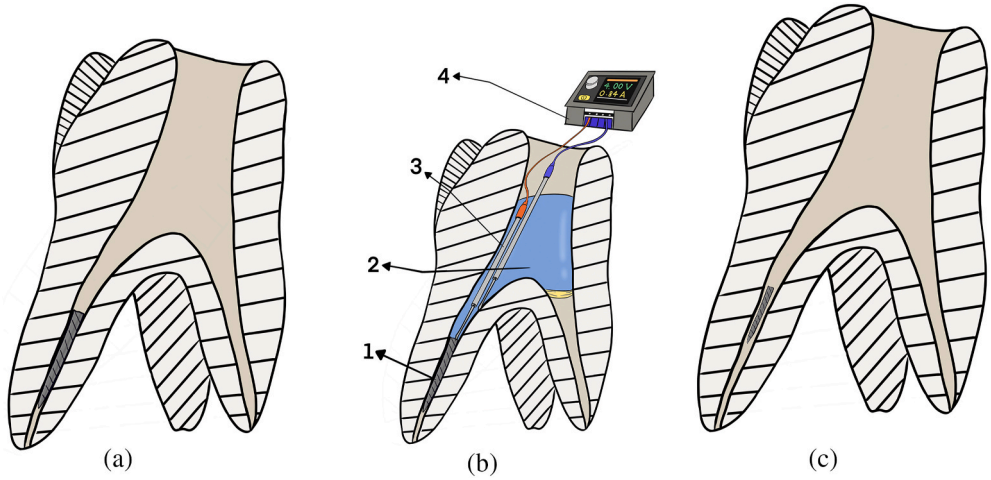

Fig. 1. Digital drawing for the electrochemical technique used in this study: A: maxillary 1st molar with fractured file in the middle third of the palatal root. B: Electrochemical technique 1. Tooth with fractured nickel-titanium (NiTi) files in the middle third, 2. The canal space is filled with fluid, 3. two electrodes inside the root canal and in direct contact with the fractured file. 4. Specially constructed electrochemical dissolution device. C. Tooth after electrochemical technique; the fractured file is smaller in size and can be easily bypassed.

electrochemical disintegration [6,7]. Fluoride-based solutions are commonly used to dissolve the electrochemical endodontic files. This approach entails active partial or total dissolution of the separated instrument by electrochemical dissolution using two electrodes (cathode and anode) in an electrolyte. Upon electric potential supply between the electrodes, the electrons migrate, releasing metallic ions into the solution [8]. Various solutions of various concentrations have been proposed [7]. In addition to the low pH (5.0) of these experimental solutions, which is a major element favouring corrosion and permitting exposure to the solution, weakening the NiTi alloy, the detrimental effect of the solution on the dentinal structure is very concerning, which might restrict its usage [8,9]. Dentin structure, such as the quantity of dentin and calcium/phosphorus (Ca/P) ratio, has been studied following the use of chemical solutions in endodontic procedures. In addition, Ni and Ti ions can be impregnated into dentinal tubules via chemical processes [8,9].

The use of fluoride-based solutions has time limitations, as they require prolonged clinical time [10] and more ions to bind to the Ni and Ti ions. Therefore, the novel solution used in this study had a new formula of NaF 12 g/L + $NaCl_2$ 60 g/L + $MgCl_2$ 60 g/L + $CaCl_2$ 60 g/L with a pH of 5 and additional ions to decrease the time required for the electrochemical dissolution of the fractured file to ensure clinical predictability with the construction of a new device, which can be used clinically for electrochemical dissolution without the need for dentin removal or use of other sensitive techniques; therefore, their effect on the hardness and calcium and phosphorus (Ca and P) ions of the dentinal following both techniques with the use of a novel chemical solution should be tested. The null hypothesis was that the use of this novel solution can affect the hardness and chemical composition of dentin when used with electrochemical and chemical dissolution techniques to manage fractured NiTi files.

2. Materials and methods

This study was approved by the ethics committee of the University of Baghdad (approval number: 637) and was conducted in accordance with the Declaration of Helsinki for both chemical and electrochemical dissolution processes with fractured ProTaper NiTi instruments (F2) (Dentsply, Switzerland) inside the canal. This study included 30 palatal roots of maxillary first molars.

2.1. Grouping

Thirty palatal roots of the maxillary first molars were used in this study and divided into three groups according to the treatment technique using the novel solution (n = 10):

Group 1: Samples treated with normal saline (control group).

Group 2: Samples treated with electrochemical dissolution.

Group 3: Samples treated with chemical dissolution.

2.2. Intracanal fragment dissolution

The sample size was calculated using a 30 percent effect in volume variation, a 25 percent standard deviation response, an alpha of 0.05, a power of 0.80, and a power analysis. Considering these assumptions, the sample size for each group was set at 10.

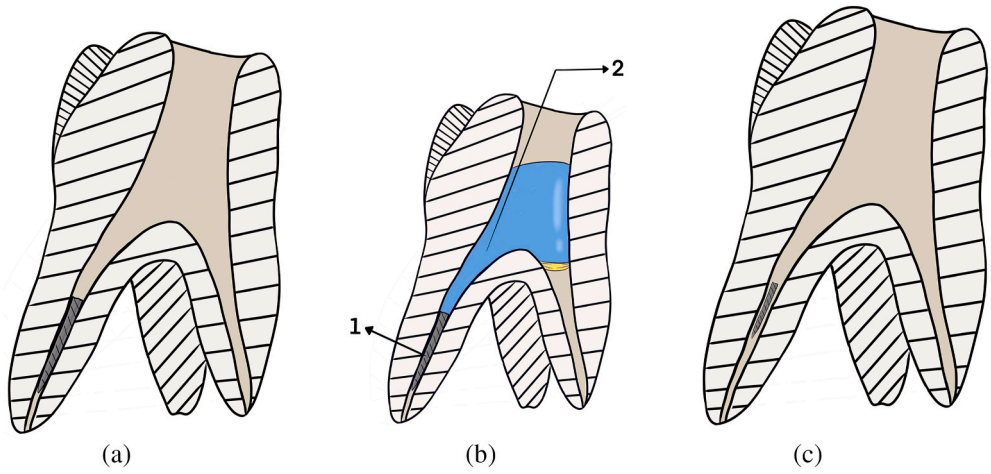

Fig. 2. Digital drawing of the chemical dissolution technique used in this study. A: maxillary 1st molar with fractured file in the middle third of the palatal root. B: Chemical technique where 1. Tooth with fractured NiTi files in the middle third, 2. The canal space is filled with fluid, C. Tooth after chemical technique, the fractured file is smaller in size and can be easily bypassed.

2.3. Sample selection and preparation

The palatal roots of the maxillary molars (n = 10) in each group used in this study met the following inclusion criteria: single canals with completely formed apices, no calcification or resorption, and roots of similar length. The exclusion criteria included endodontically treated teeth and teeth with cracks [11]. The teeth were stored in 10 % formalin solution until needed. To eliminate any remaining formalin, the teeth were washed under running water [12]. The teeth were initially radiographed, and the pulp chamber was opened with continuous irrigation using diamond burs. A size 08 K-file (Dentsply Sirona, Ballaigues, Switzerland) was passively placed inside the palatal root canal up to the apical foramen to assess foramen patency and measure the root canal length, which varied from 20 to 22 mm [13]. To weaken the F2 instrument, a diamond bur was used 2.5 mm from the instrument tip [14].

Each instrument was inserted with constant apical pressure into the palatal root canal and spun at 300 rpm using an electric motor (X-smart, USA) until the tool fractured and the fragment was inserted into the root canal (Fig. 1 A) [10]. The position of each piece in the middle third of the root canal was verified using radiographs of the teeth following fracture. An experienced endodontist used a size 08 K file to determine whether the file could bypass the fractured file. To validate the site of the fragment in the root's middle third, a post-fracture radiograph was taken [10].

The teeth were pressed into a rubber stopper within a hole pre-drilled in a vial to avoid direct manipulation. A rubber dam sheet was placed over the roots of the vial's root [12]. To form a reservoir for the solution, each tooth's dental crown was attached to an acrylic microcell, which had an internal volume of 516 mL and an internal diameter of 9.12 mm^3 during the dissolving procedure [10].

This study employed 600 mL of a novel solution (NaF 12 g/L + $NaCl_2$ 60 g/L + $MgCl_2$ 60 g/L + $CaCl_2$ 60 g/L) with pH 5.0 based on the formulation reported in a previous pilot study. The solution was placed in a microcell for both the chemical and electrochemical techniques. In the electrochemical technique, the intracanal fragment was touched with the tip of a 0.2 mm platinum wire (China) [10], which was confirmed by radiography. The working electrode (WE) was composed of a platinum wire. The reference (RE) and counter (CE) electrodes were connected by a second gold wire, 0.5 mm diameter). Each wire was submerged in the acrylic microcell solution and connected to +4.0 V potential provided by a special electrical device (Fig. 1B), which was connected to an ammeter measurement gauge that can register the anodic current reaction during the test, as shown in Fig. 1C, for 10 min to cause partial dissolution of the NiTi file to easily bypass it, based on the spectrophotometer (Hamilton, Japan) test for dissolution of both Ni and Ti in relation to time.

During the chemical procedure (Fig. 2A), the chemical solution was placed inside the canal for 30 min (Fig. 2B), dissolution decreased with time with increased electrical charge generated during the dissolution procedure. Therefore, new chemical solution was added every 15 min. This technique required 30 min based on a spectrophotometer test for the dissolution of both Ni and Ti in relation to time. The same endodontist passively inserted a size 08 K-file into the root canal to check whether the fragment could be bypassed (Fig. 2C).

2.4. Vickers microhardness test

The external surfaces of the palatal roots were longitudinally grooved using a diamond disc in a straight micromotor handpiece at a speed of 1500 rpm with a steady flow of water coolant [15]. With a chisel and hammer, each root specimen was broken into two segments; the segment with the fewest surface aberrations on dentin was selected for the test and horizontally inserted into an auto-polymerising acrylic disc.

The surface hardness of the root dentin was determined using a digital Vickers hardness tester with a 100 g load for 15 s [16]. The

Table 1
Descriptive statistics of the Vickers hardness number (VHN) and analysis of variance (ANOVA) test for the experimental groups.

Descriptive statistic					ANOVA	
Group	Level	N	Mean VHN	Std. Deviation	P-value	Significance
Control	Coronal	10	65.33	0.69929	<0.001	HS
	Middle	10	62.28	0.56725		
	Apical	10	55.33	0.80561		
Electrochemical	Coronal	10	65.25	0.8182	<0.001	HS
	Middle	10	62.14	0.63105		
	Apical	10	55.01	0.3414		
Chemical	Coronal	10	65.19	0.86852	<0.001	HS
	Middle	10	62.09	0.48865		
	Apical	10	55	0.8165		

$P < 0.001$; HS, highly significant.

Table 2
Tukey's test for the tested techniques.

Tukey HSD						
Dependent Variable			Mean Difference (I-J)	Std. Error	P-value	Sig
Control	Coronal	Electrochemical	−7.18000*	0.22179	0.000	HS
		Chemical	−10.09000*	0.22179	0.000	HS
	E	Chemical	−2.91000*	0.22179	0.000	HS
Electrochemical	Coronal	Electrochemical	−7.10000*	0.17097	0.000	HS
		Chemical	−10.13000*	0.17097	0.000	HS
	E	Chemical	−3.03000*	0.17097	0.000	HS
Chemical	Coronal	Electrochemical	−7.16000*	0.26612	0.000	HS
		Chemical	−10.09000*	0.26612	0.000	HS
	E	Chemical	−2.93000*	0.26612	0.000	HS

$P < 0.001$, HS, highly significant.

Table 3
Analysis of variance (ANOVA) test for the coronal, middle, and apical third of the teeth for the tested techniques.

ANOVA							Sig
		Sum of Squares	Df	Mean Square	F	P-value	
Coronal	Between Groups	0.002	2	0.001	0.006	0.994	NS
	Within Groups	4.861	27	0.180			
	Total	4.863	29				
Middle	Between Groups	0.041	2	0.020	0.099	0.906	NS
	Within Groups	5.533	27	0.205			
	Total	5.574	29				
Apical	Between Groups	0.013	2	0.006	0.018	0.983	NS
	Within Groups	9.754	27	0.361			
	Total	9.767	29				

$P < 0.001$.

indentation was performed using a diamond indenter, and the indented area was scanned using a microscopic lens (400 ×) connected to a Vickers tester. Microhardness measurements were performed for each sectioned root at three separate locations (coronal, middle, and apical thirds; 3, 6, and 9 mm from the root tip) [17,18], and an indentation was made on the dentin surface. The representative hardness value at each point was obtained as the average of three indentations to produce a single value. All readings were obtained by the same examiner using the same calibration machine.

2.5. Energy dispersive spectroscopy

Sputter-coated samples were prepared, and imaging and microanalysis were performed on a Bruker Nano (X flash detector 410- M, Germany) at 3 keV; three measurements were performed for each root canal at 3, 6, and 9 mm from the apex (nine measurements for each root canal) [9].

2.6. Statistical analysis

The data were analysed using SPSS (version 25, IBM Corporation, USA), and the statistical analysis distribution was checked using

Table 4
Descriptive statistics of the Ca & P concentrations and Ca/P ratio in the teeth before and after chemical and electrochemical dissolutions.

Descriptive Statistics					Ca/p	Mean	Standard Deviation
		N	Mean	Std. Deviation			
Ca	Control	10	4.584	0.00516	Control	1.0854	0.00182
	E	10	4.581	0.00738			
	CH	10	4.579	0.00738	E	1.08	0.00219
p	Control	10	4.225	0.00527			
	E	10	4.223	0.00483	CH	1.0843	0.00168
	CH	10	4.223	0.00483			

Ca, Calcium; P, Phosphate; E, Electrochemical; CH, Chemical.

the D'Agostino–Pearson normality test. One-way analysis of variance (ANOVA) and Tukey tests ($P < 0.05$) were employed to compare the hardness of the teeth before and after both the electrochemical and chemical dissolution tests because they had a normal distribution (Table 1).

Interfacial analytical tests were performed for the coronal, middle, and apical thirds of the teeth using each technique to determine any statistical differences before and after the dissolution techniques (Tables 1 and 2). In addition, interfacial analytical tests were performed between the coronal, middle, and apical thirds of the teeth using each technique to determine the statistical differences between the tested techniques in each subgroup (Table 3).

Analysis of variance (ANOVA) was used to analyse the correlation of means between groups, and Tukey's test for multiple parametric comparisons was performed.

3. Results

Table 1 shows the microhardness values for teeth obtained after the dissolution of the fractured instrument by electrochemical and chemical dissolution techniques with the control group. The hardness of the tooth did not change significantly ($P > 0.05$) because of the dissolving procedures.

Tables 1 and 2 show the results of ANOVA and Tukey's test, respectively, for the tested (9, 6, and 3 mm) coronal, middle, and apical thirds of the teeth in each technique, which demonstrated highly significant differences ($P < 0.05$) between the groups in the three areas.

Table 4 represents the amount of Ca and P ions in the teeth of the three groups and shows the amount of Ca/P ions in the teeth before and after performing the chemical and electrochemical dissolution techniques.

4. Discussion

The null hypothesis was rejected as no alteration was observed in the Vickers hardness number between the control and experimental groups following both the electrochemical and chemical dissolution techniques, with no change in the chemical composition of dentin. Determination of the microhardness of teeth provides indirect evidence of a change in the mineral content of dental hard tissues [19]. Thus, microhardness determination is the initial step in evaluating dentin behaviour under stress [19]. This study was designed to verify the effects of chemical and electrochemical dissolution techniques on tooth microhardness in the management of fractured NiTi files in the palatal roots of human maxillary molars.

The results confirmed the main premise, as shown in Tables 1 and 3, which indicated that the dissolution procedure did not cause a significant loss in dentin hardness, with no significant differences from the control group. The novel solution [$NaF + NaCl_2 + MgCl_2 + CaCl_2$] with a pH of 5.0 used in this study, acted by partially dissolving the NiTi instrument components due to its acidity and ions contained in the solution, which acted mainly on Ni ions to restore the original canal. Furthermore, the solution demonstrated low cytotoxicity when tested on human fibroblasts in another study [9] and required less time to bypass the fractured file, demonstrating the ability to be used in a simple chemical dissolution technique. Moreover, the solution can be used with electrochemical techniques for a short clinical duration demonstrating promising results, as confirmed in a pilot study.

$CaCl_2$ and $MgCl_2$ were added to promote Ni dissolution because Ca^+ and Mg^+ have a strong reactivity with Ni in the presence of hydrogen [20], which increases the dissolution process. In addition, the actions of Na^+, F^+, and Cl^- on Ti at pH 5 play an important role. Although the chemical solution used for dissolution contained NaF, with the possibility of fluoride absorption by the root, this did not occur. This may be attributed to the presence of the smear layer, which could have inhibited the absorption of the fluoride (12,000 ppm) that was in contact with the dentin walls for 30 min according to a previous study [21]. Mineralised collagen matrix residues, endodontic sealants, and root canal irrigants are commonly observed in the smear layer [22]. In addition, the smear layer of the root canal walls may be protected by forming a barrier [23]. Buchalla, Lennon, Becker, Lucke, and Attin (2007) investigated whether the smear layer had an impact on the dentinal absorption of fluoride at 12,000 ppm with pH 4.0 [24]. According to their findings, the presence or absence of a smear layer interferes with fluoride absorption in bovine dentinal specimens. The buffering action of dentin should also be highlighted when it comes to pH. Macedo et al. investigated the differences when dentin was exposed to sodium hypochlorite concentrations at various pH values for 1 h, to a pH 5.0 NaOCl solution which was discovered to potentially act as a buffer [25].

Application Note

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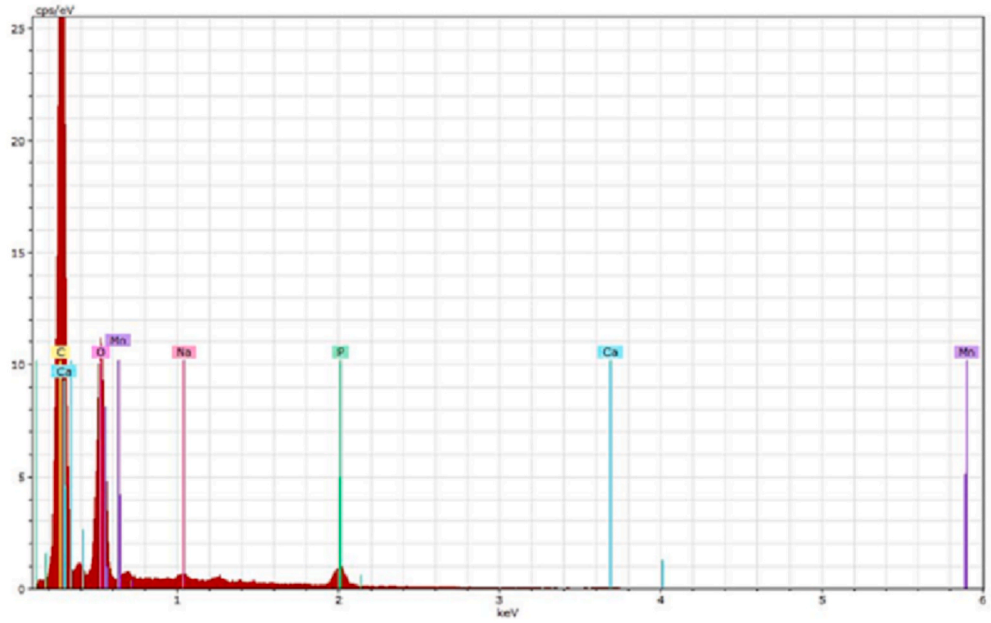

Spectrum: Acquisition 5183

El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
Ca	20	L-series	58.38	58.38	34.58	13.84
O	8	K-series	18.46	18.46	27.40	3.09
C	6	K-series	17.09	17.09	33.78	2.71
P	15	K-series	4.22	4.22	3.24	0.34
Mn	25	L-series	1.52	1.52	0.66	0.70
Na	11	K-series	0.33	0.33	0.34	0.07
		Total:	100.00	100.00	100.00	

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Fig. 3. Energy-dispersive X-ray of the dentinal tubules of the teeth after electrochemical dissolution.

′When NiTi files are inserted into electrolytes, the instrument acts as an electron conductor and the electrolyte (novel solution) as a medium for electrical current transport; thus, the electrode reaction describes the phase boundary reaction equivalent to the charge exchange between the instrument and electrolyte [26]. For the electrochemical technique, the file attached by the WE and immersed in

the novel solution in the presence of another electrode gold wire with diameter 0.5 mm was connected to CE + RE immersed in the same solution. Both the electrodes were attached to specially designated device to control voltage to 4 V for 10 min and can register the anodic current reaction during the test; thus, in the electrolyte solution, positively charged ions of metal movement will be towards the anodic partial reaction leaving the electrons with a negative charge in the metal (i.e., the electron conductor), indicating oxidization of the metal atoms. In the cathodic partial reaction, the dissolved metal ions at the phase boundary gather electrons and deposit themselves as metal atoms on the electrode surface [27].

In this study, a significant regional difference was observed between the coronal and apical thirds of the teeth (Tables 2 and 3). The microhardness decreased apically, which may be explained by the histology of the relative characteristics of the apical dentin and the patterns of the root canal dentin. Ballal et al. (2010) [28] found that microhardness increased from the apical to the coronal thirds. According to Inoue et al. (2013) [29], mechanical properties are proportional to the tissue mineral content, and coronal dentin contains more minerals than radicular dentin; coronal dentin hardness was approximately 0.8 GPa, which is higher than radicular dentin hardness.

The EDX test of the teeth revealed the presence of different chemical elements (Fig. 3), and multiple comparison tests were performed between the groups, which did not show statistically significant differences. The Ca/P ratio remained practically constant (Table 4). Further studies are warranted to confirm the effect of this novel solution and dissolution technique on the tooth microhardness and Ca/P ratio in dentin to confirm the reliability of the results.

5. Conclusion

No effect on the dentinal microhardness and alteration in the Ca/P concentration were observed following treatment of the fractured NiTi file in the extracted teeth using the novel chemical solution with both the chemical and electrochemical techniques.

6. Funding and conflict of interest

This research project was self-funded.

7. Data availability statement

The data supporting the findings of this study have not been deposited in a publicly available repository. The data are available upon request.

CRediT authorship contribution statement

Linz Ali Shalan conceived the presented idea, developed the theory, performed computations, and verified the analytical methods and writing. **Hussain F. Al-Huwaizi** supervised the methodology and findings of this study and wrote, reviewed and edited the manuscript.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.heliyon.2024.e35902.

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