Comparative analysis of various corrosive environmental conditions for NiTi rotary files

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ABSTRACT

The aim of the present study is to compare the corrosion tendency using two kinds of NiTi files in the various environmental conditions through the visual examination and electrochemical analysis. ProTaper Universal S2, 21 ㎜ (Dentsply Maillefer, Ballaigues, Switzerland) and Hero 642, 0.06 tapers, size 25, 21 ㎜ (Micromega, Besancon, France) rotary instruments were tested. The instruments were randomly divided into eighteen groups (n = 5) by the immersion temperature, the type of solution, the brand of NiTi rotary instrument and the presence of mechanical loading. Each file was examined at various magnifications using Scanning Electron Microscope (JEOL, Akishima, Tokyo, Japan) equipped with energy dispersive X-ray microanalysis (EDX). EDX was used to determine the components of the endodontic file alloy in corroded and noncorroded areas. The corrosion resistance of unused and used NiTi files after repeated uses in the human teeth was evaluated electrochemically by potentiodynamic polarization test using a potentiostat (Applied Corrosion Monitoring, Cark-in-Cartmel, UK).

Solution temperature and chloride ion concentration may affect on passivity of NiTi files. Under the conditions of this in vitro study, the corrosion resistance is slightly increased after clinical use.
[J Kor Acad Cons Dent 33(4):377-388, 2008]

Key words : NiTi file, Corrosion, Loading, Chloride, Temperature

- Received 2008.4.14., revised 2008.5.16., accepted 2008.6.9.-

Ⅰ. INTRODUCTION

The goal of root canal treatment is to achieve a high standard of disinfection of the root canal system through chemo-mechanical instrumentation[1,2]. with the uses of sodium hypochlorite (NaOCl) and Nickel-Titanium (NiTi) rotary instruments[3,4].

While the introduction of NiTi rotary instruments into endodontics has provided the benefit with characteristics of super-elasticity and high strength, their separation during root canal shaping has been a great concern[5]. NiTi rotary instruments usually fracture through two mechanisms: torsion or metal fatigue caused by flexure[6,7]. Fracture caused by fatigue failure mechanism occurs due to crack initiation on the cutting surfaces and propagation toward the file's axial cen-

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ter[8-10].

Recently, the influence of NaOCl on the corrosion of NiTi rotary instruments has been studied[11]. It is known that NaOCl, a chlorine-containing solution, is corrosive to metals. Corrosion adversely affects the metallic surfaces by causing pitting and porosity, and decreases the cutting efficiency of endodontic files. Pitting corrosion is initiated by localized chemical or mechanical damage to the protective oxide film. It is supposed that the micropitting acts as microstructural defects for the crack formation and stress risers[12]. Also, corrosion fatigue may be initiated at the base of corrosion pits. Corrosion-fatigue is the result of the combined action of alternating or cycling stresses and corrosive environment[13]. The fatigue process is thought to cause rupture of the protective passive film, upon which corrosion is accelerated.

It is well known that NiTi alloys have corrosion resistance thanks to the surface film of titanium oxide[14]. The passive film acts as protective coating to protect the underlying surface from further chemical reaction, such as corrosion, electrodissolution, or dissolution. The passivation of metal is affected by many factors like solution temperature, composition of material, and chloride ion concentration.

The influence of NaOCl on the corrosion of NiTi rotary instruments has been studied and the results by various authors are contradictory[15-19]. Significant signs of surface corrosion were reported by O'Hoy and colleagues[20] after overnight immersion, whereas Darabara *et al.*[21] found no signs of corrosion after immersion for 1 hour in 5.25% NaOCl and 17% ethylenediaminetetraacetic acid (EDTA) heated to 37℃. The inconsistent results are due mainly to the different study conditions and interpretations. There is no report about the corrosion of the NiTi rotary instruments after chemo-mechanical preparation and cleaning procedure using NaOCl solution in the root canals of human teeth.

Therefore, the purpose of the present study is to evaluate the corrosion resistance of used and unused NiTi files in the various environmental conditions through the microstructural observation of optical and Scanning Electron Microscpoe (SEM) examination and electrochemical analysis.

Ⅱ. MATERIALS AND METHODS

1. Material preparation

A total of 45 ProTaper Universal S2, 21 ㎜ (Dentsply Maillefer, Ballaigues, Switzerland) and 45 Hero 642, 0.06 tapers, size 25, 21 ㎜ (Micromega, Besancon, France) rotary instruments were tested.

The instruments were randomly divided into eighteen groups (n = 5). Two groups (HC and PC) were assigned as the control group (negative control group; no exposure to any environment) and sixteen groups (H1~H8 and P1~P8) were divided by the conditions such as immersion temperature, the type of solution, the brand of NiTi rotary instrument and the presence of mechanical loading (Table 1).

Extracted fifty maxillary and mandibular human molars were used to simulate clinical conditions. The irrigating solutions used were commercial 5.25 wt% NaOCl and distilled water; both solutions were prepared just before instrumentation. The instruments were driven by an electronic motor (X-smart; Dentsply Maillefer, Ballaigues, Switzerland) set at 300 rpm and torque of 2.4 N. The mechanical preparation of 20 root canals for each file was done without lubricant at room temperature (21 ± 2℃).

After shaping each canal, the files were placed in a separate glass vials filled with 10 ml of the 5.25 w% NaOCl solution or distilled water for overnight (18 h). Vials were either stored at room temperature (22℃) or were placed in an electronically controlled heating device (Multiblock 2001; Labline, Mehare Park, IL, USA) after the preset immersion temperature of 55℃ had been reached. Before microstructural evaluation, the files were cleaned in an ultrasonic bath with distilled water and air dried.

Table 1. Groups for experimental conditions

Group	Brand of file	Storage medium	Immersion temperature (°C)	Mechanical stress
HC	Hero	-	-	-
PC	ProTaper	-	-	-
H1	Hero	Distilled water	22	-
H2	Hero	Distilled water	22	+
H3	Hero	Distilled water	55	-
H4	Hero	Distilled water	55	+
H5	Hero	NaOCl	22	-
H6	Hero	NaOCl	22	+
H7	Hero	NaOCl	55	-
H8	Hero	NaOCl	55	+
P1	ProTaper	Distilled water	22	-
P2	ProTaper	Distilled water	22	+
P3	ProTaper	Distilled water	55	-
P4	ProTaper	Distilled water	55	+
P5	ProTaper	NaOCl	22	-
P6	ProTaper	NaOCl	22	+
P7	ProTaper	NaOCl	55	-
P8	ProTaper	NaOCl	55	+

Table 2. Scoring system for evidence of corrosion[19)*]

Score	Criteria
0	no corrosion
1	Mild corrosion (surface pitting with no discoloration)
2	Moderate corrosion (pitting with corrosion products)
3	Severe corrosion (pitting corrosion and metal separation)

[19)] Stockes *et al.*

2. Optical and SEM examination for corrosion

Files from the control groups (unused) and experimental groups were examined for evidence of corrosion using the criteria of shown in Table 2. Precipitated corrosion products were identified as a precipitate within the solution or deposited on the metal surface.

After optical microscope (OPMI pico; Carl zeiss, Obercohen, Germany) assessment of corrosion, each file was examined at various magnifications using Scanning Electron Microscope (JEOL, Akishima, Tokyo, Japan) equipped with energy dispersive X-ray microanalysis (EDX). EDX was used to determine the components of the endodontic file alloy in corroded and noncorroded areas.

3. Statistical analysis

Statistical analysis of the collected data was performed with SAS version 9.1. Log-linear model

(Poisson regression) was used to exam the effect of explanatory variable (experimental conditions) to dependent variable (score) (Table 5). The differences revealed in the data were designated as significant at $p < .05$.

4. Potentiodynamic polarization test

The corrosion resistance of unused and used NiTi files after repeated uses in the human teeth was evaluated electrochemically by potentiodynamic polarization test using a potentiostat (Applied Corrosion Monitoring, Cark-in-Cartmel, UK). A platinum wire was employed as a counter electrode. The reference electrode employed was an Ag / AgCl electrode with an electrochemical potential of -16 mV with respect to the saturated calomel electrode (SCE). All potentials in the text refer to the SCE, which is +242 mV with respect to the normal hydrogen electrode (NHE). The NaOCl solution was heated to 37℃ and this temperature was held until the end of the tests. Polarization curves were obtained with a potential scan rate of 10 mV / sec.

Ⅲ. RESULTS

1. Optical microscopic evaluation

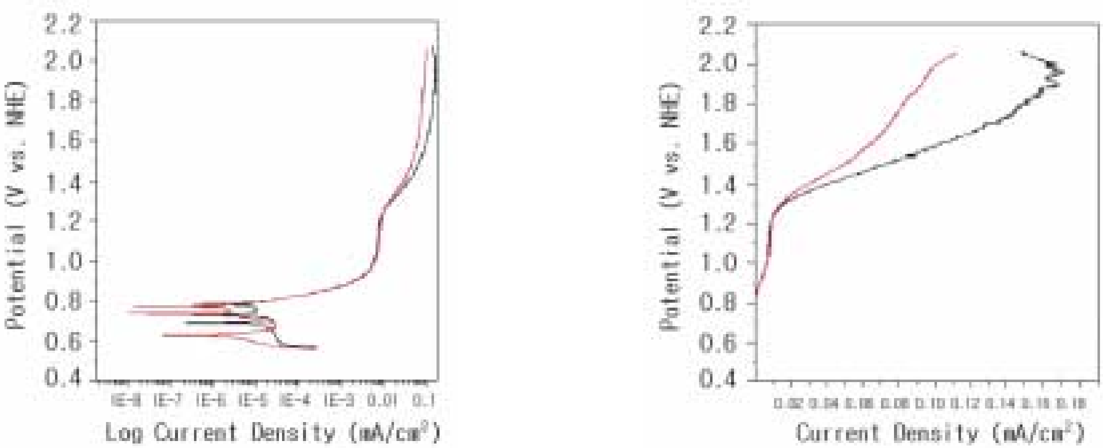
The results of each group were summarized in Table 3 and Table 4. No obvious sign of corrosion was found on the flutes of all experimental files when immersed in distilled water and NaOCl solution at room temperature (22℃). On the other hand, general corrosion signs like changes of metal color and existence of precipitate were observed when immersed in NaOCl solution at heating temperature (55℃). This phenomenon

Table 3. Frequency of corrosive scores in each group (n = 5)

Group	Corrosion Criteria			
	No (0)	Mild (1)	Moderate (2)	Severe (3)
HC	5	0	0	0
PC	5	0	0	0
H1	5	0	0	0
H2	5	0	0	0
H3	2	3	0	0
H4	2	3	0	0
H5	5	0	0	0
H6	5	0	0	0
H7	0	0	5	0
H8	0	0	4	1
P1	5	0	0	0
P2	5	0	0	0
P3	3	2	0	0
P4	3	2	0	0
P5	3	2	0	0
P6	3	2	0	0
P7	0	0	5	0
P8	0	0	5	0

(A) (B) (C) (D)

Figure 1. Corrosion appearance of ProTaper and Hero 642.
A; ProTaper in NaOCl at 55℃ B; Hero 642 in NaOCl at 55℃ C; ProTaper after immersing in NaOCl (left) and distilled water (right) at 55℃ D; Hero 642 after immersing in NaOCl (left) and distilled water (right) at 55℃.



(A) (B)

Figure 4. Representative potentiondynamic polarization curves of tested instruments; red line - new file and black line - used file.

Table 4. The corrosive scores at each condition (mean ± S.D.)

Brand of file	Storage medium	Immersion temperature (℃)	Mechanical stress	mean ± S.D.	group
Hero	Distilled water	22	-	0.00 ± 0.00	H1
			+	0.00 ± 0.00	H2
		55	-	0.60 ± 0.55	H3
			+	0.60 ± 0.55	H4
	NaOCl	22	-	0.00 ± 0.00	H5
			+	0.00 ± 0.00	H6
		55	-	2.00 ± 0.00	H7
			+	2.20 ± 0.45	H8
ProTaper	Distilled water	22	-	0.00 ± 0.00	P1
			+	0.00 ± 0.00	P2
		55	-	0.40 ± 0.55	P3
			+	0.40 ± 0.55	P4
	NaOCl	22	-	0.40 ± 0.55	P5
			+	0.40 ± 0.55	P6
		55	-	2.00 ± 0.00	P7
			+	2.00 ± 0.00	P8

Table 5. Comparison of environmental conditions for NiTi file corrosion

Analysis of Parameter Estimates				
Parameter	Estimate	Standard Error	Chi-Square	P-value
Intercept	0.7715	0.2423	10.14**	0.0015
Brand of file	-0.0364	0.2697	0.02	0.8927
Storage medium	-1.5041	0.3496	18.51***	〈.0001
Immersion Temperature	-2.5455	0.5192	24.03***	〈.0001
Mechanical stress	-0.0364	0.2697	0.02	0.8927

** : $p < .01$, *** : $p < .001$

Table 6. Comparative effectiveness of corrosive conditions

Storage medium	Immersion temperature (℃)	Fitted value
Distilled water	22	0.036
	55	0.464
NaOCl	22	0.164
	55	2.086

The more corrosion was detected in the groups with high temperatures and NaOCl immersion and the lesser with low temperature and Distilled water ($p < .05$).

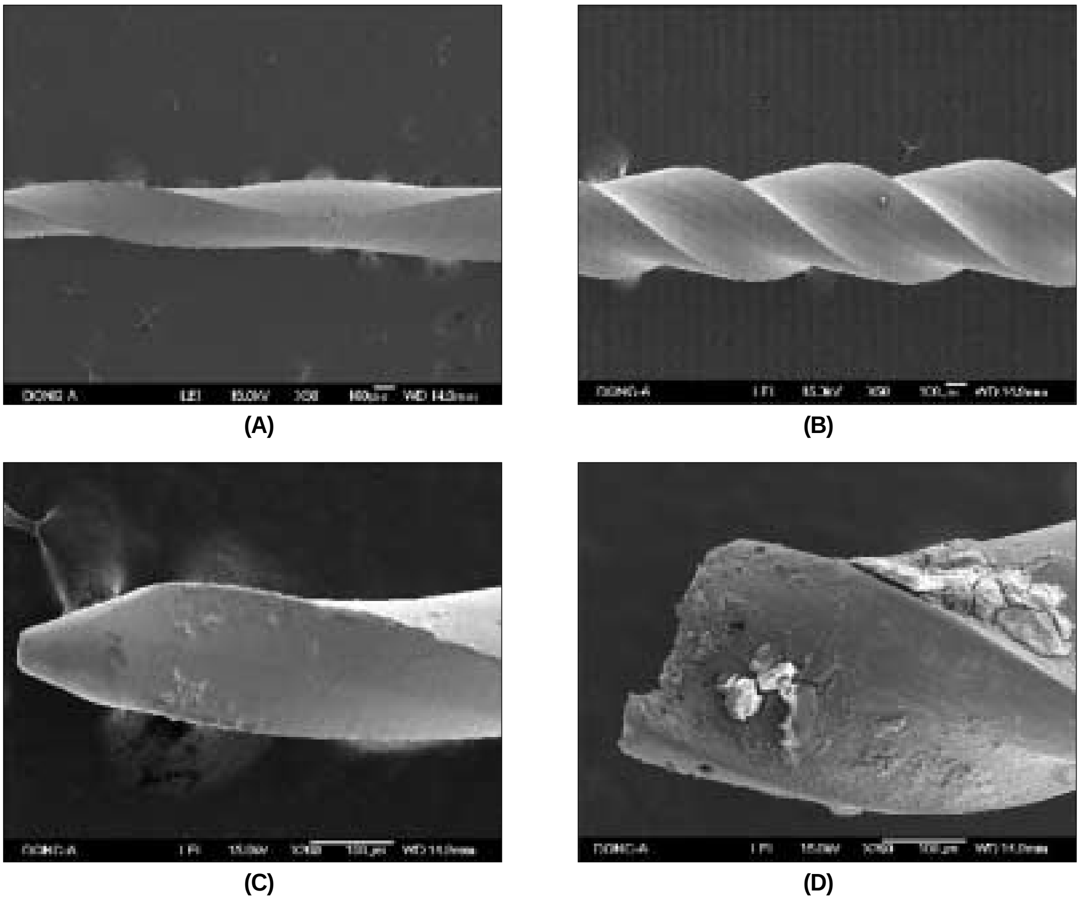


Figure 2. SEM observation of NiTi files as representative of Group P2 (A), Group H5 (B), Group P8 (C), and Group H8 (D).

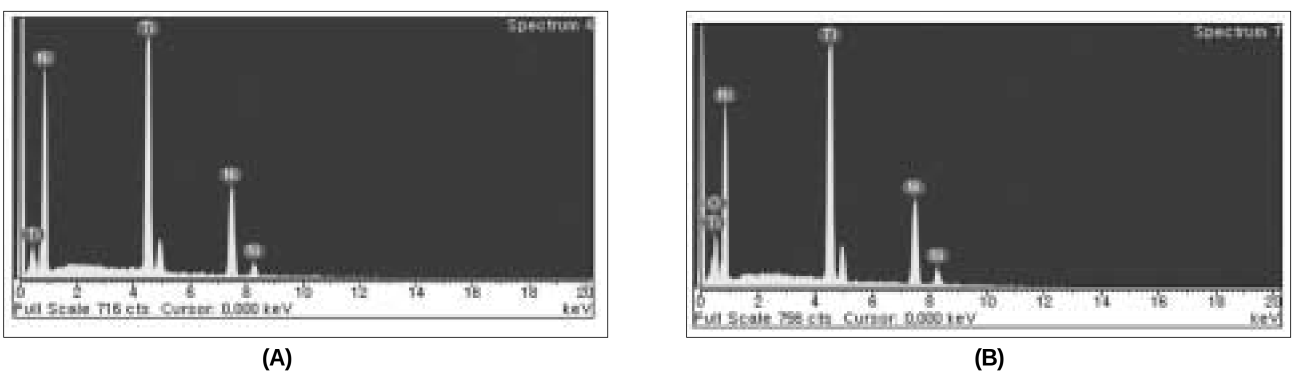


Figure 3. (A) EDX spectra of the Hero 642 instrument shown in Figure 2 B. The presence of Ni and Ti on the non-corroded areas is clearly seen. (B) EDX spectra of the Hero 642 instrument shown in Figure 2 D. Oxides are shown in corroded areas.

was more evident in the ProTaper files which have metal shaft than in the Hero 642 files which have plastic shaft (Figure 1).

Through the statistical analysis using Log-linear model, among the four environmental conditions of corrosion, the storage media and the immersion temperature had affected significantly to corrosion ($p < .0001$) (Table 5).

After application to Log-linear model fitted offset ($y = e^{0.7354 - 1.5041 \times \text{storage} - 2.5455\, t \times \text{temp}}$), the higher fitted value was detected in the groups with high temperatures and NaOCl immersion (Table 6).

2. Scanning electron microscope with energy dispersive X-ray microanalysis

SEM showed typical patterns of torsional and fatigue failure (Figure 2A). SEM analysis of the files exposed to the room temperature NaOCl solution indicated the same morphology for all the files without any evidence of localized corrosion (Figure 2B). No difference was observed in comparison to the files as received. The degradation effect of immersion in heated NaOCl solution was clear as localized attacks both on the cutting edges and on the flat surface in the SEM images (Figure 2C and D). Data analysis showed that immersion temperature and presence of dissimilar metals had significant effects on the corrosion of NiTi rotary files. The EDX spectra revealed the similar components between the noncorroded and corroded areas of NiTi files (Figure 3).

3. Electrochemical analysis

Figure 4 (A) presents typical polarization curves of used and unused NiTi files in NaOCl reagent as logarithmic scale of current density. It can be observed that the polarization curves of two specimens showed similar results. After conjecturing the result, used file (black line) insignificantly demonstrated rather higher corrosion potential as 0.7 V than unused file (red line) (0.6 V).

Figure 4 (B) shows results of the same experiment as algebraic scale of current density. In this figure, used file indicates higher corrosion potential in excessively high potential region above 1.2 V, what is called the region of breakdown of passivity.

Ⅳ. DISCUSSION

Pitting corrosion causes metal failure by the creation of small holes in metal surfaces. Fracture caused by fatigue failure mechanism occurs due to crack initiation at the cutting surfaces[22]. It is generally associated with specific species in solution, by far the most common of which is chloride. Some sorts of metals are more intrinsically resistant to corrosion than others, either due to the fundamental nature of the electrochemical processes involved or due to the details of how reaction products form[23]. It is well known that titanium shows high corrosion resistance in an alloy to chloride compound[24]. Nevertheless, with the presence of chloride ions, some microscopic breakdown events can be observed on titanium at potentials far below the pitting potential.

Given the right conditions, a thin film of corrosive products can form on metal's surface spontaneously, acting as a barrier to further oxidation. Passivation is extremely useful in alleviating corrosion damage. NiTi alloys are generally passivated by the surface film of titanium and nickel oxide. The corrosion patterns of NiTi alloy involve selective removal of nickel from the surface[25]. Thus, it may be that after some initial dissolution of nickel from the surface, the nickel-titanium alloy may form a surface containing mainly titanium oxides in the outer layer and nickel-titanium in the inner layer[26,27]. Surface oxidation seems to be very promising for improving the corrosion resistance and biocompatibility of NiTi[28].

Certain conditions, such as high concentrations of chloride, can interfere with a given alloy's ability to re-form a passivating film. The growth of passivating titanium oxide film involves migration of oxide ions through oxide anion vacancies. On reaching the metal / film interface, new oxide is formed at that interface. In the presence of chloride ions, it can migrate in parallel across the passivating oxide. Thus, if chloride ions reach the metal / film interface, it would form the metal chloride. If sufficient metal chloride were to accumulate there, the oxide would rupture explosively, and reveal a bare metal surface, forming a microscopically saturated chloride solution. The oxide film requires sufficient chloride accumulation at one specific spot in order for film rupture to occur.

Raising the temperature generally causes metals to pit more readily. Pit nucleation and metastable propagation have not been much examined in terms of temperature dependence. The effects of solution temperature and chloride ion concentration on the electronic properties of passive film on pure metals have been investigated for many years[29]. Chloride ions migrate across the passivating oxide film faster at higher temperatures. It is supposed that the concentration of oxygen and metal vacancy in the passive film has increased accompanying with it an increase of temperature and chloride ion concentration. It was noticed that the instruments immersed in heated NaOCl solution from this study produced marked visible dark particles in the solution as evidence of corrosion. Heating NaOCl solutions up to 60°C has been suggested to increase reactivity and antimicrobial and tissue-dissolving action[30]. However, the increase in reactivity may also enhance the corrosive potential of commercial NaOCl solutions and adversely affect mechanical properties of NiTi rotaries. Such an effect of heating of NaOCl on NiTi file integrity was described by Peters *et al*[31]. They found a reduction in fatigue resistance after immersion in NaOCl solution for two hours at 60 ℃, not only for machined ProFiles but also for electropolished RaCe rotaries.

There were differences in visual corrosion appearance after immersion between unused files and used files in human teeth from this study. The potentiodynamic polarization test was done to evaluate stress factor through comparing the corrosion resistance between unused and used files in human teeth. Generally, corrosion potential value is indicative for the ionization tendency of materials in specific media. The ionization tendency is decreased towards higher corrosion potential values. There are reports suggesting that NiTi alloy cannot re-passivate itself speedily if the surface film is disrupted or damaged after contacting against the root canal wall[32]. However, in the present study, used files after contacting against the root canal wall several times insignificantly demonstrated rather higher corrosion potential values than unused files. According to the results of this study, NiTi files seem to have the ability to re-form the passive film after repeated clinical uses.

The other type of metal corrosion is galvanic corrosion. Galvanic corrosion occurs when two different metals electrically contact each other and are immersed in an electrolyte[33]. ProTaper files showed more severe corrosion appearance than hero 642 files in NaOCl solution. EDX microanalysis showed that the cutting and non-cutting sections of the ProTaper file were made of a NiTi alloy, whilst the shank was made of gold-plated brass. Otherwise, the shank of the hero 642 file was non metallic material. Thus the NiTi alloy seemed to act as the anode and the gold-coated shaft acts as the cathode in the corrosion cell when the instrument was immersed in NaOCl solution. Galvanic corrosion also occurs independently in some cases. NaOCl may be present in the pulp chambers of teeth with restorations of different metals (amalgam, gold, etc.) and NiTi files could be damaged mechanically by various metals. If the passive film is damaged mechanically, the spot acts as the anode and the other parts of the passive film acts as the cathode. When a galvanic couple forms, one of the metals in the couple becomes the anode and corrodes faster than it otherwise would, whilst the other becomes the cathode and corrodes more slowly than it would alone. One important factor influencing galvanic corrosion is area ratios of the anode and cathode[34]. A small anode / cathode area ratio is highly undesirable. In this case, the galvanic current is concentrated onto a small anodic area. Rapid loss of thickness of the dissolving anode tends to occur under these conditions.

As for most metallic materials, the localized corrosion behavior of NiTi alloy depends on environmental conditions and it is very hard to analyze quantitatively. EDX spectra represent qualitative analysis of the corroded region. The presence of oxides on the corroded areas in the EDX spectrum could not be the firm evidence of corrosion because of passive film products. Moreover, the verification of stress corrosion cracking and corrosion fatigue usually requires specific equipments. Further investigation will be necessary to relate corrosion and unexpected cracks in NiTi rotary endodontic instruments more closely.

Ⅴ. CONCLUSIONS

Under the condition of this in vitro study, the solution temperature and chloride ion concentration had an effect on passivity of NiTi files. In addition, corrosion performance of NiTi files could be increased by galvanic corrosion with presence of dissimilar metal.

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국문초록

니켈티타늄 파일의 부식에 영향을 미치는 다양한 환경 조건 비교

염지완 · 박정길 · 허 복 · 김현철*
부산대학교 치의학전문대학원 치과보존학교실

이 연구에서는 다양한 부식 환경하에서 니켈티타늄 파일에 대한 부식 경향을 전기화학적 검사와 시각적 검사를 이용해 비교 평가하였다.

각 45개의 21 ㎜ ProTaper Universal S2와 21 ㎜ #25/0.06의 Hero642 file을 기계적 하중 여부, 저장 용액(증류수 및 차아염소산나트륨), 저장 온도(22℃ 및 55℃), 파일 종류에 따라 18개의 그룹(n = 5)으로 나누었다. 40개의 발거 치아 근관을 성형한 후 18시간 동안 저장 용액에 조건에 따라 보관하였다. 부식 산물의 생성 여부나 변색 등을 관찰하고 주사전자현미경을 이용해 각 시편을 다양한 배율에서 관찰하였으며 에너지 분산형 X선 분광기로 성분 분석을 하였다. 전기화학적분극시험을 통하여 새 파일과 근관 성형에 사용한 파일의 부식저항성을 검사하였다.

본 실험 조건하에서는 저장 용액의 종류와 보관 온도가 니켈 티타늄 파일의 부식에 영향을 주는 것으로 나타났다. 또한 이종 금속이 존재하는 경우 갈바닉 부식의 영향으로 인해니켈 티타늄 파일의 부식 경향이 증가할 수도 있음을 보였다.

주요어: 니켈티타늄 파일, 부식, 하중, 염화물, 온도