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Influence of sodium hypochlorite on fracture properties and corrosion of ProTaper Rotary instruments

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Abstract

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Aim To evaluate the influence of immersion in NaOCl on resistance to cyclic fatigue fracture and corrosion of ProTaper NiTi Rotary instruments.

Methodology A total of 120 new ProTaper NiTi Rotary files (F2) were randomized and assigned to three different groups of 40 each. Group 1 was the control group; 20 mm (excluding the shaft) of group 2 instruments were immersed in 5% NaOCl at 50 °C for 5 min; instruments in group 3 were completely immersed in 5% NaOCl at 50 °C for 5 min. All instruments were then tested for cyclic fatigue, recording the time in seconds to fracture. Data were analysed by the Kruskall–Wallis test and *post-hoc* multiple comparisons ($P < 0.05$). Micromorphological and microchemical analyses were also completed by means of a field emission scanning electron microscopy (SEM) on those instruments in group 3 that had undergone early fracture.

Results Instruments in group 3 had a significantly lower resistance to fracture because of cyclic fatigue than those in groups 1 and 2 ($P < 0.001$). In some instruments in group 3, early fracture occurred after only a few seconds of fatigue testing. SEM observations revealed evident signs of corrosion of the fractured instruments.

Conclusion Group 3 had significantly reduced resistance to cyclic fatigue compared with instruments in groups 1 and 2. The phenomenon of early fracture may be attributed to galvanic corrosion induced by the presence of dissimilar metals, where one acts as the cathode of a galvanic couple, established when the instrument is immersed in NaOCl solution. The NiTi alloy may acts as the anode and thus undergoes corrosion.

Keywords: cyclic fatigue, fracture, nickel-titanium, rotary instruments, sodium hypochlorite.

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Introduction

The goals of root canal treatment are to achieve a high standard of disinfection of the root canal system through chemo-mechanical instrumentation and fill the canal to prevent re-infection (Schilder 1967, 1974). Canal preparation requires a continuous and progressively tapered shape, so as to allow sodium hypochlorite (NaOCl) to be delivered to the apical section of canal and perform its bactericidal action (Shih *et al.* 1970, Bystrom & Sundqvist 1983, 1985, Rutala 1990, Dychdala 1991, Best *et al.* 1994, Rutala & Weber 1997) and to dissolve organic substances (Grey 1970, Hand *et al.* 1978, Koskinen *et al.* 1980). The introduction of NiTi instruments into endodontics has provided the profession with rotary instruments of greater taper, with characteristics of super-elasticity and high strength (Walia *et al.* 1988). It has been shown that NiTi Rotary instruments can achieve excellent taper, with less risk of canal transportation

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and improved tooth-structure preservation, and do so more rapidly than hand files (Glosson *et al.* 1995, Thompson & Dummer 1997, Portenier *et al.* 1998, Gluskin *et al.* 2001, Peters *et al.* 2001, Pettiette *et al.* 2001). However, various factors may cause unexpected fracture during clinical use of NiTi Rotary instruments (Kosa *et al.* 1999). Instruments that rotate in a curved canal are subjected to cyclic fatigue caused by repeated tensile-compressive stress (Pruett *et al.* 1997, Kuhn & Jordan 2002, Berutti *et al.* 2003). It also appears that cyclic fatigue has a cumulative effect on an instrument, weakening it over time until fracture (Ruddle 2001). Obviously this is an important matter due to the influence that a broken instrument, if left in place, can have on the long-term outcome of root canal treatment (Manasse & Britto 2000), as it can impede cleansing and shaping of the root canal system.

NiTi instruments come into contact with NaOCl during disinfection (Savage & Walsh 1995) or when the solution is present in the pulp chamber and root canal during instrumentation (Bystrom & Sundqvist 1983). It is known that NaOCl is corrosive to metals. The corrosion patterns, involving selective removal of nickel from the surface, can create micropitting (Sarkar *et al.* 1983). It is supposed that these microstructural defects can lead to areas of stress collection and crack formation, weakening the structure of the instrument (Oshida *et al.* 1992). The influence of NaOCl on the corrosion of NiTi instruments has been studied (Busslinger *et al.* 1998, Haikel *et al.* 1998, Stokes *et al.* 1999); despite minor signs of corrosion being detected, they did not appear to involve clinically significant alteration of the mechanical properties and performance of the instruments. O'Hoy *et al.* (2003) evaluated the effect of cleaning procedures using NaOCl, and detected significant corrosive phenomena of NiTi instruments exposed to 1% NaOCl for up to 10 cleaning cycles. However, no significant reduction of torque at fracture or number of revolutions to flexural fatigue was found.

The hypothesis evaluated in this *ex vivo* study is that possible corrosive phenomena, triggered by contact between metals with different electrochemical activities in the presence of NaOCl, may alter the structural integrity of the surface of a NiTi instrument and thus, in critical fatigue conditions, predispose it to fracture. The primary objective was to evaluate the effect of immersion in NaOCl solution on the resistance of ProTaper F2 Rotary instruments to cyclic fatigue fracture. The secondary objective was to analyse those instruments that underwent unusual fracture patterns (premature fracture) micromorphologically and microchemically, using a field emission scanning electron microscope (SEM) equipped with a dispersion energy microprobe, in order to characterize the alloys and the corrosion products. The *null hypothesis* tested was that there would be no significant difference ($\alpha = 0.05$) between the three groups in terms of time to breakage under standard experimental conditions.

Materials and methods

Cyclic fatigue testing

A total of 120 ProTaper F2 Rotary instruments (Dentsply Maillefer, Ballaigues, Switzerland), all from the same production lot, were randomly assigned (using a random numbers table) to three different groups of 40 each.

In group 1 (control group, $n = 40$) the instruments were not immersed in NaOCl. In group 2 ($n = 40$) the instruments were immersed to a depth of 20 mm (thus excluding the gold-coated shaft of the instrument), for 5 min in 5% NaOCl at 50 °C. In group 3 ($n = 40$) the instruments were completely immersed for 5 min in 5% NaOCl at 50 °C. The instruments in groups 2 and 3 were then washed for 1 min with distilled water and air-dried.

All instruments in all three groups were then subjected to tests of resistance to fracture because of cyclic fatigue, using an instrument (Fig. 1) specifically developed for the purpose (Maillefer SA, Ballaigues, Switzerland) and already used in previous studies (Fife *et al.* 2004). The apparatus was linked to an endodontic

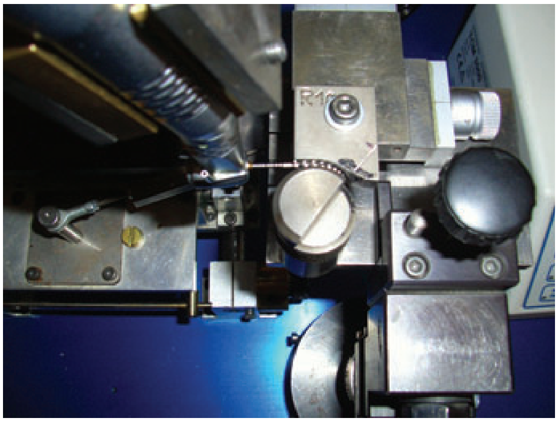

Figure 1 ProTaper F2 instrument inserted in the cyclic fatigue test apparatus (Maillefer SA).

motor and enabled the instrument to rotate freely within a stainless steel artificial canal at a constant pressure. The engine was set to 300 rpm. All instruments were rotated at constant speed inside the artificial canal, which had 60° curvature and 10 mm radius of curvature. During the test cycle, each instrument was cooled with constant air flow to avoid overheating. For each instrument, the time in seconds from the start of the test until the moment of breakage was recorded with a chronometer. The Kolmogorov–Smirnov for normality test revealed a non-normal distribution in group 3. For this reason the differences amongst groups were analysed with the nonparametric Kruskall–Wallis (K–W) test and the *post-hoc* multiple comparisons between groups were performed by the Mann–Whitney *U*-test. The differences were considered statistically significant when $P < 0.05$, adjusted for the number of comparisons.

Field emission SEM observations

Instruments that demonstrated unusual fracture patterns (premature fractures) were observed through a field emission SEM equipped with a dispersion energy microprobe (Leo Ultra 55 FEG SEM equipped Oxford Inca EDX system, acceleration voltage: 0.1–30 kV, probe current: 4 pA–10 nA, gun: field emission gun; LEO Electron Microscopy, Inc., Thornwood, NY, USA) in order to characterize alloy and corrosion products.

Results

Descriptive statistics are summarized in Table 1 and Fig. 2. In group 3 some unusual premature instrument fractures occurred, at very low cyclic fatigue values (7 s), as is clear in Table 1 and Fig. 2, which gives minimum and maximum instrument failure times.

The inferential analysis revealed statistically significant differences between the three groups (K–W = 15.68; df = 2; $P < 0.001$). *Post-hoc* analysis revealed a significantly lower resistance to cyclic fatigue in group 3 ($P < 0.001$ for both comparisons) than in either group 1 or group 2; the latter two groups were not significantly different ($P = 0.42$). The degradation effect of immersion in NaOCl solution is clear in the SEM images (Figs 3 and 4). The surface of the rotary instrument in the control group showed no localized attack or microcracks (Fig. 3), whilst immersion in the solution (group 3) caused localized attacks both on the cutting edges and on the flat surfaces (Fig. 4, a). The enlargement (Fig. 4, b) evidences the pitting and microstructural alterations in the area around the cavity; as expected, the EDS spectra revealed the presence of Ni and Ti on the noncorroded

Table 1 Descriptive statistics: case summaries for instrument failure time (s)

Group	*n*	Mean (s)	Median	Standard deviation	Standard error of mean	Min	Max
1	40	142.47	139.0	25.42	4.02	87	190
2	40	136.50	142.5	21.29	3.37	90	177
3	40	105.15	123.5	50.04	7.91	7	186

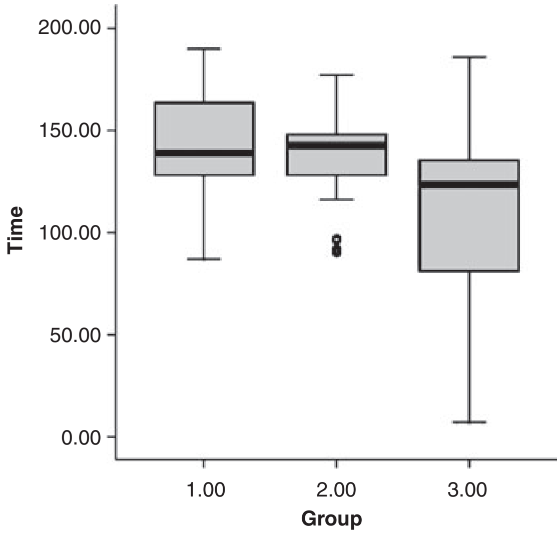

Figure 2 Box–Whisker plot of the instruments failure time (s) by groups.

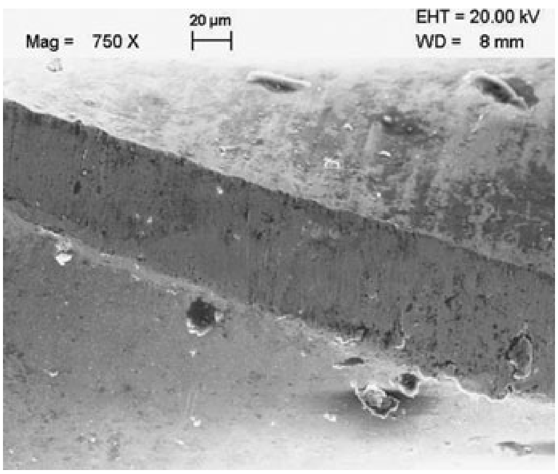

Figure 3 Photomicrograph of a ProTaper F2 file (control group). The surface of the rotary instrument shows no localized attack or microcracks.

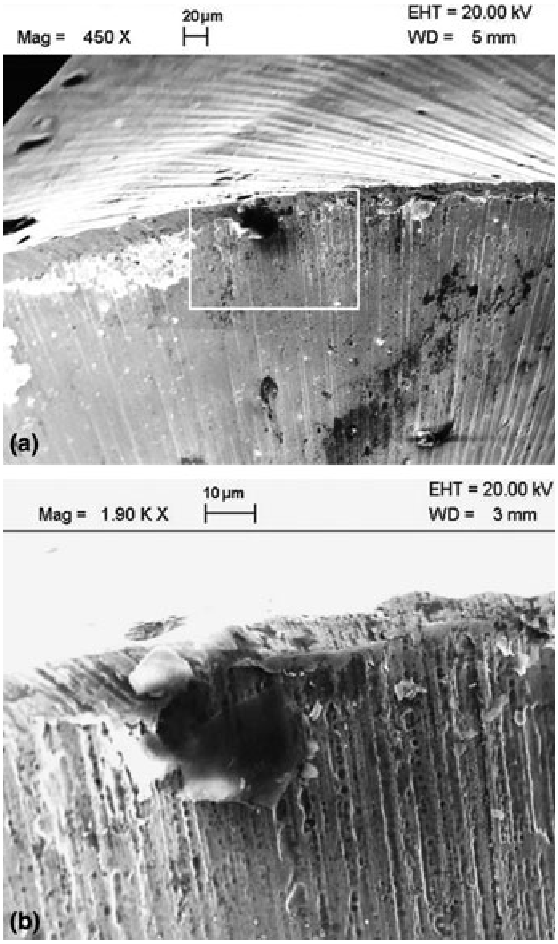

Figure 4 (a) Photomicrograph of a ProTaper F2 instrument after complete immersion in sodium hypochlorite solution (group 3). The image shows localized attacks both on the cutting edges and on the flat surfaces. (b) The enlargement reveals the pitting and microstructural alteration of the area around the cavity.

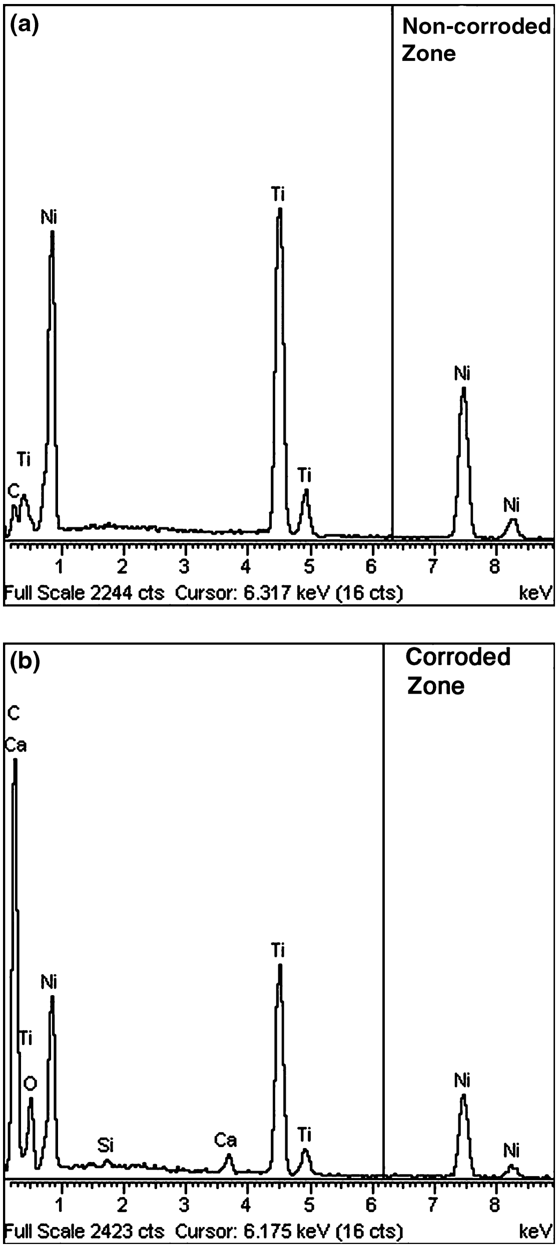

Figure 5 (a) EDS spectra of the ProTaper F2 instrument shown in Fig. 4a. The presence of Ni and Ti on the noncorroded areas is clear. (b) Whilst in the corroded zones oxides are evidenced.

areas (Fig. 5, a), whilst in the zones where localized attacks took place, the corrosion products were identified as oxides of Ni and Ti together with calcium carbonate coming from the tap water employed for the solutions (Fig. 5, b). Figure 6 shows the fracture surface of an endodontic instrument; the fracture proceeds intergranularly, the localized corrosion attacks being preferential sites for the growth and propagation of cracks.

Discussion

The ProTaper System (Dentsply Maillefer) is a relatively recent NiTi rotary system which was designed to provide cutting efficiency and flexibility (West 2001, Ruddle 2002) in a kit with few instruments. It consists of three shaping files (SX, S1 and S2) which shape the coronal and the middle thirds of the root canal, and three finishing files (F1, F2 and F3) designed to shape the apical portion of the root canal. A layer of gold alloy

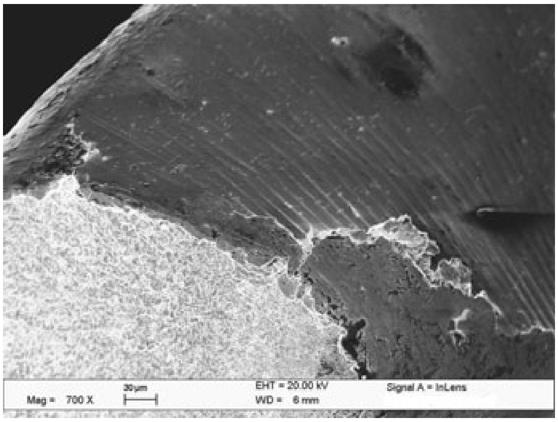

Figure 6 Photomicrograph of the fracture surface of ProTaper F2 instrument (group 3). The fracture proceeds intergranularly and the localized corrosion attacks are preferential sites for the growth and propagation of cracks.

is electro-deposited on the shaft of these instruments for the purpose of distinguishing them from other instruments made by the same manufacturer, by their colour.

This study used the ProTaper F2 because it has been shown to possess lower resistance to fracture because of cyclic fatigue than the other instruments in the ProTaper series (Fife *et al.* 2004). It is known that instruments with low taper have lower resistance to torsional stress, whereas those with greater size and taper are more subject to fracture because of cyclic fatigue (Pruett *et al.* 1997, Sattapan *et al.* 2000). Instruments in group 3, in which the files had been completely immersed in NaOCl, showed a significantly lower resistance to cyclic fatigue fracture compared with the other two groups. Furthermore, some instruments in group 3 unexpectedly underwent premature fracture, even though they were subjected to the same test conditions as the other instruments. It was also noticed that the instruments in this group, at the moment of immersion in NaOCl, produced marked effervescence in the solution, with formation of visible dark particles in suspension. This was attributed to the different metals present in the ProTaper instruments tested that, in the presence of an electrolytic solution such as NaOCl, can set off galvanic reactions and corrosion processes. In clinical practice it is very unlikely that an instrument would be completely immersed in NaOCl, as the shaft is completely lodged within the head of the endodontic handpiece. However, it should be considered that sodium hypochlorite may be used as a disinfectant agent during cleaning procedures of the instruments after their use and that NaOCl may be present in the pulp chambers of teeth with restorations of different metals (amalgam restorations, gold crowns, etc.). Both conditions might trigger galvanic reactions and thus corrosion processes.

The field emission SEM images and EDS spectra clearly revealed the presence of noncorroded zones and of corroded areas where localized attack had created pitting and cracks close to the fracture surface. The phenomena observed may be attributed to galvanic corrosion, also known as 'dissimilar metal corrosion', i.e. corrosion induced when two (or more) dissimilar metals are coupled in a corrosive electrolyte (Angelini *et al.* 2002). It occurs when two dissimilar materials are brought into electrical contact under water. 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. When contact with a dissimilar metal is made, however, corrosion rates change: corrosion of the anode accelerates and corrosion of the cathode decelerates or even stops. The driving force for corrosion is the potential difference between the different materials. A small anode/cathode area ratio is highly undesirable, as in this case the galvanic current is concentrated onto a small anodic area. Rapid thickness loss of the dissolving anode tends to occur under these conditions. Galvanic corrosion cells can be set up on the macroscopic level or on the microscopic level. On the microstructural level, different phases or other microstructural features can be subject to galvanic currents.

The integrity of dental restorations and intraoral prostheses may be compromised because of galvanic corrosion (Fraker & Griffin 1985, Angelini *et al.* 2002). Damage to dental restorations and prostheses because of a failure to understand the corrosive properties of the various couples that are set up between different materials have been reported in the restorative and implant fields (Angelini *et al.* 1988, Rosalbino *et al.* 2003). In the case of the rotary endodontic instruments under study, consisting of a Ni-Ti alloy with a gold-coated shaft, the instrument itself acts as the anode in the corrosion cell that originates when the instrument is immersed in NaOCl solution, and the gold-coated shaft acts as the cathode.

In the original study design, instruments in groups 2 and 3 were immersed for 5 min in NaOCl. Considering that the mean life of instruments in group 1 (control) in the test conditions was approximately 2 min 30 s, in agreement with Fife *et al.* (2004), it was decided to double the contact time of the instrument with the solution, so as to emphasize its effect whilst remaining within realistic times for clinical practice. In this study 5% NaOCl was used at 50 °C to simulate the clinical situation, because it has been shown that a higher temperature increases its capability to dissolve organic substrate (Cunningham & Balekjan 1980). Furthermore, used at 50 °C, NaOCl contributes to reducing the deposit of smear layer left after manual instrumentation (Berutti & Marini 1996).

Other studies have considered the effects of NaOCl on NiTi alloys. Haikel *et al.* (1998) used hand instruments immersed in a 2.5% solution for 24–48 h, and detected no obvious corrosion. This is probably the result of a very slow corrosion of NiTi alloy alone, when the galvanic effect does not potentiate it, as occurs where other metals are present. O'Hoy *et al.* (2003) found greater corrosion in the shafts of rotary instruments tested, and hypothesized that the cause of corrosion lay in the different nature of the metal alloy of the shaft coating. Busslinger *et al.* (1998) used 5% NaOCl for 30 or 60 min and Lightspeed rotary instruments, and found corrosion patterns, even if the authors were not sure of the clinical implications.

Conclusions

Within the limitations of the study, it may be concluded that if NiTi rotary instruments operate immersed in a NaOCl solution contained in the pulp chambers of teeth restored with metals or alloys having different electrochemical nobility values, galvanic corrosion may occur. These coupling phenomena may cause pitting and cracks that alter the integrity of the instrument surface, decreasing its resistance to fracture because of cyclic fatigue. The location of the pitting caused by corrosion appears to be random, which might explain the occurrence of unexpected and unpredictable premature fracture. The clinical implication is that the use of NaOCl as disinfectant when cleaning nickel-titanium instruments whose shaft is coated with different metals should be carefully considered. Furthermore, the arising question should be whether in the presence of metallic crown restorations, it may be preferable to use irrigants with lubricating properties and that do not favour the formation of galvanic currents. This hypothesis requires to be investigated in greater depth in further studies.

Obviously, sodium hypochlorite must be used for the appropriate time to achieve cleansing of the canal system, but this may be carried out after the use of rotary NiTi instruments has been completed.

Further investigation will be necessary to relate galvanic corrosion and unexpected cracks in Ni-Ti rotary endodontic instruments more closely. Electrochemical tests are now underway to quantify the galvanic currents in NaOCl solutions at different temperatures, and will include microstructural characterization.

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