RESEARCH ARTICLE

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Comparative Evaluation of Cyclic Fatigue and Corrosion Resistance of Three Different Nickel-Titanium Instruments

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Abstract

Objective: The aims of the study are to investigate the effect of the surface treatment process on prolonging the life of the rotary instruments against cyclic fatigue and the effect of the surface coating process on the corrosion resistance of Ni-Ti files.

Methods: This study was carried out in two stages. In the first one, a total of 120 Ni-Ti rotary instruments (25/.04 taper) from 3 brands (HeroShaper, RaCe, and TF) were selected. Sixty of them were anodized then all of them were tested in simulated root canals with two different curvatures. The time until fracture was recorded, and the number of cycles was calculated for each group. The second stage; the effect of anodization on corrosion resistance was assessed. The file groups tested in 5% NaOCl solution at 37° C. Data were subjected to The Mann-Whitney U test. The significance level was set at P= 0.05.

Results: There was a statistical difference among the rotary systems in terms of cyclic fatigue. Among the file types, TF instruments were the most resistance to cyclic fatigue and RaCe instruments had the lowest results. The effect of anodization process was different among file groups.

Conclusion: Within the limits of this study, the increase in canal curvature reduces the life of the rotary instruments. While the anodization process did not change cyclic fatigue in all file groups in 20° curved canals, it reduced the wear life of RaCe and HeroShaper files and extended the wear life of TF files in canals with 37° curved canals.

Key words: Anodization, Canal curvature, Corrosion, Cyclic fatigue, HeroShaper, RaCe, Ni-Ti, Rotary system files, Twisted File.

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INTRODUCTION

The root canals must be cleaned, shaped, disinfected, and hermetically filled for successful endodontic treatment (1). Recently, nickel-titanium (Ni-Ti) rotary instrument systems have been used for shaping root canals. These systems are exposed to cyclic fatigue and corrosion in the root canal. Sudden breakage of files because of this stress affects the success of endodontic treatment negatively. Metal fatigue results from cyclic fatigue, static torsional fatigue, and dynamic torsional fatigue. Cyclic fatigue is the common cause of breakage in continuous rotary systems (2-4).

In parallel with the developments in the field of engineering, various surface treatments are applied to the files to prolong the life and increase its durability. Among these applications, the most frequently used process is surface coating with anodization. Anodization is controlled oxidation performed to form a protective oxide film or layer on a metal surface that can slow or prevent further oxidation (5).

Effective canal irrigation along with mechanical preparation is also very important for the success of endodontic treatment. Besides their antimicrobial benefits, irrigation solutions also facilitate mechanical shaping. Despite all these advantages, these solutions can have some chemical and electrochemical effects on the canal instruments, causing corrosion on the surface of the canal files, which is manifested by pitting and porosity (6). The studies have reported that the pits and porosity formed on the surface because of corrosion reduce the cutting capacity of the file and decrease the resistance against breakage (6,7). Therefore, it is desirable that Ni-Ti canal files are not affected by the irrigation solution.

The aims of this study are to investigate the effect of the surface treatment process on prolonging the life of the rotary instruments against cyclic fatigue and corrosion tendency of Ni-Ti files and the effect of the surface coating process on the corrosion resistance of Ni-Ti files. The first null hypothesis is that there are not significant differences among the instruments in terms of the number of cycles to file breakage and time to fracture. The second null hypothesis is that the surface treatment process does not affect the life of the rotary instruments against cyclic fatigue. The third null hypothesis is that the surface treatment process does not affect the corrosion resistance of Ni-Ti files.

METHODS

This experimental study is taken from the thesis "Comparative Evaluation of Cyclic Fatigue and Corrosion Resistance of Three Different Nickel-Titanium Instruments" done in 2011 at Atatürk University. This study was approved by the Ethics Committee of the graduate school of health sciences of Atatürk University, Erzurum, Turkey, (2011.3.1/7).

This study was carried out in two stages. In the first stage, the cyclic fatigue test was applied to covered and non-covered RaCe (FKG Dentaire, La-Chaux-de-Fonds, Switzerland), HeroShaper (Micro-Mega, Besancon, France), and Twisted Files (TF) (SybronEndo, Orange, CA). The second stage; the corrosion resistance of these systems in 5% NaOCl was evaluated.

Cyclic fatigue test.

The cyclic fatigue testing block prepared by Atatürk University Engineering Faculty was used in this study. The properties of the canals on the testing block were 16-mm the working length, 5-mm the radius of curvature, and 20°, 26°, 30°, and 37° the angles of curvature, 1 mm the diameter and depth. The canals have 20° and 37° angles of curvatures were used in this study.

Before the test procedures, surface images were taken from the canal files with Scanning Electron Microscopy (SEM) (JSM 6400-Scanning Electron Microscope, Jeol, Tokyo, Japan).

Then, a total of 60 files (20 files from each system) were randomly divided into 6 groups.

- 1. Group: 25/.04 RaCe files (20° angle of curvature) (n=10)
- 2. Group: 25/.04 HeroShaper files (20° angle of curvature) (n=10)
- 3. Group: 25/.04 TF files (20° angle of curvature) (n=10)
- 4. Group: 25/.04 RaCe files $(37^{\circ}$ angle of curvature) (n=10)
- 5. Group: 25/.04 HeroShaper files (37° angle of curvature) (n=10)
- 6. Group: 25/.04 TF files (37° angle of curvature) (n=10).

X-Smart (Dentsply, Maillefer, Ballaigues, Switzerland) torque-controlled endodontic motor was used with 2 N/cm torque and 500 rpm

The lubricant gel (FileCare gel; Munich, Germany) was applied to the testing canals for each file. The chronometer, 1/100 second sensitivity, was started when the file started to rotate, and it stopped

when the file broke. The total working time was recorded for each file.

The lubricant on the surface of the broken files was cleaned by an ultrasonic cleaner. Then, SEM images were taken from the broken surfaces.

Anodization (Anodic Oxidation) process

The coating process was carried out at 25°C, in 2 M acetic acid solution, at 10 V current, for 5 minutes and the surface images were taken by SEM. Then, a total of 60 files (20 files from each system) were randomly divided into 6 groups.

- 1. Group: anodized 25/.04 RaCe files (20° angle of curvature) (n=10)
- 2. Group: anodized 25/.04 HeroShaper (20° angle of curvature) (n=10)
- 3. Group: anodized 25/.04 TF (20° angle of curvature) (n=10)
- 4. Group: anodized 25/.04 RaCe (37° angle of curvature) (n=10)
- 5. Group: anodized 25/.04 HeroShaper (37° angle of curvature) (n=10)
- 6. Group: anodized 25/.04 TF (37 $^{\circ}$ angle of curvature) (n=10)

All the cyclic fatigue procedures mentioned above were done for these groups as well.

In the corrosion test, potentiodynamic scanning was performed using the Gamry G750 (Pennsylvania, USA) block in the Engineering Faculty of Atatürk University.

Corrosion process

A triple corrosion unit consisting of canal file, reference electrode, and counter electrode was set up in a glass container. The reference electrode was Ag/AgCl, and the counter electrode was graphite. After connecting the file and electrodes (according to

the recommendations of the manufacturer), measurements were performed at a scanning speed of 2 millivolts/sec in the potential range of -1 volt / +1 volt.

5% NaOCl (White Med, U.K.) was used as solution and the solution temperature was kept constant at 37°C, which is body temperature throughout the procedure. HeroShaper, RaCe, and TF files, and anodized HeroShaper, anodized RaCe, and anodized TF files were subjected to the corrosion test. The obtained data were recorded.

Statistical analysis

The descriptive analyses for the groups were calculated. The Mann-Whitney U test was performed after the normality and homogeneity tests. The data were analyzed using Statistical Package for the Social Sciences (SPSS Inc., Chicago, IL, USA) for Windows, version 15 software. The statistical significance was set at p < 0.05.

RESULTS

The results of non- covered HeroShaper, RaCe, and TF files in 20° curved experimental setup are shown in table 1. There were significant differences among the instruments in terms of the number of cycles to file breakage and time to fracture in 20° curved experimental setups (Table 2).

Table 1. The means and standard deviations (SD) of the time and the number of cycles to file breakage in 20° curved experimental setup.

scrup.				
	The time to file	The number of		
	breakage			
	mean ±SD	breakage		
		mean ±SD		
HeroShaper	457.66±62.39	3813.83±519.90		
RaCe	375.39±55.21	3128.23±464.27		
TF	814.99±249.04	6791.61±2075.34		

Table 2. *P* values among the groups in 20° curved experimental setups. (p < 0.05)

setups. $(p < 0.05)$				
Instruments	HeroShaper	RaCe	TF	

HeroShaper	-	0.008	0.000
RaCe	0.008	-	0.000
TF	0.000	0.000	-

Results of non-covered HeroShaper, RaCe, and TF files in 37° curved experimental setup are shown in table 3. There were significant differences between HeroShaper and RaCe (p=0.000). and between TF and RaCe (p=0.000) in terms of the number of cycling to file breakage and time to fracture (p>0.05) (Table 4). However, there were no significant differences between HeroShaper and TF (P=0.121) (Table 4).

Table 3. The means and standard deviations (SD) of the time and the number of cycles to file breakage in 37° curved experimental setup.

setup.		
	The time to file breakage mean ±SD	The number of cycles to file breakage mean ±SD
HeroShaper	64.39±6.43	536.58±53.55
RaCe	31.02±5.42	258.51±45.22
TF	71.9±12.41	599.19±103.44

Table 4. *P* values among the groups in 37° curved experimental setup (p < 0.05).

instruments	HeroShaper	RaCe	TF
HeroShaper	-	0.000	0.121
RaCe	0.000	-	0.000
TF	0.121	0.000	-

Evaluation of the effect of root canal curve

The increase of root curve in the experimental setup was caused significant decrease in the number of cycling and time to file breakage for all instruments (Table 5).

Table 5. P values between 20° and 37° non-covered curved canals (p < 0.05).

	HeroShape	RaCe	TF
	r		
P (time)	0.000	0.000	0.000
P (number of cycles)	0.000	0.000	0.000

Evaluation of the Effect of Anodization Process

There was no significant effect of the anodization process statistically among the groups for 20° curved experimental setups. The P values are shown in Tables 6.

Table 6. *P* values between covered and non-covered instruments in 20° curved canals.

			HeroShaper	RaCe	TF
P (time)		1.000	0.45	0.07
P	(number	of	1.000	0.45	0.07
cyc	eles)				

There was significant effect of the anodization process statistically among the groups for 37° curved experimental setups. The p values are shown in Tables 7.

Evaluation of Corrosion Test

As a result of the corrosion test, it was found that the anodization process had a positive effect on the HeroShaper file group.

In RaCe and TF gruops, the anodization process reduced the corrosion resistance of the files. Especially for RaCe files, this amount of reduction is greater than others. For TF files, on the other hand, the values are very close for non-covered files. (Table 8)

Table 7. *P* values between covered and non-covered instruments in 37° curved canals.

		HeroShaper	RaCe	TF
P (time)		0.000	0.001	0.041
P (number cycles)	of	0.000	0.001	0.041

Table 8. Corrosion test results (E corr: corrosion potential, I corr: corrosion current)

	HeroShaper (non-covered)	HeroShaper (anodized)	RaCe (non-covered)	RaCe (anodized)	TF (non-covered)	TF (anodized)
E corr	-237.0 mV	42.40mV	414 mV	-310 mV	-119 mV	-216 mV
I corr	4.020 uA	14.80 uA	132.0 nA	335.0 nA	801.0 nA	1.070 uA

Evaluation of SEM Images of files

Non-covered HeroShaper and TF files showed traces of manufacturing on their surfaces, while surfaces of RaCe files were smooth. Since the electropolishing process is performed during the manufacturing phase of RaCe files, possible burrs and roughness are eliminated from the file surface. All three file types have particles on them, and especially TF files have bubble-shaped images. The reason for this image is due to differences in the manufacturing stage. Unlike other Ni-Ti files, TF files are produced with R-Phase heat treatment technology. Although

this heat treatment creates a porous structure in the metal, it is a production method that increases the durability of the metal. This is also supported by our fatigue test results (Figure 1).

Both crusting and smearing appearances were formed, due to the plastic deformation during cyclic fatigue. The crack is marked and obviously it occurred due to the high load. When the breaking section was examined, it was observed that a spongy structure was formed. Crack starting points are evident. (Figure 2A)



Figure 1. Image of instruments before test procedures A: HeroShaper, B: RaCe, and C: TF Investigation of Broken Surfaces Non-covered HeroShaper Files



Figure 2. Image of non-covered instruments after test procedures A: HeroShaper, B: Race, and C: TF

Broken Surface Investigations of non-covered RaCe Files

Traces of plastic deformation are evident on the edges of the file. The crack started from the sharpest point of the cutting-edge part of the file. This suggests that geometrical properties of files in edge designs may be important in terms of metal fatigue.

When the breaking section is examined, it is seen that the crack progresses in the form of tearing. The initial crack origin is smooth. The smoothness of the surface disappeared in the final breaking zone. (Figure 2B)

Broken Surface Investigations of non-covered TF Files

No traces of deformation were observed in the breaking section. The pores from the manufacturing are evident on the surface and most likely caused initiation of the crack (Figure 2C).

Broken Surface Investigations of Covered HeroShaper Files

The breaking images after coating are similar to the breaking images of non-covered files. Again, traces of plastic deformation are evident in the breaking sections. Porous structures were formed on the surface due to the coating, which caused a decrease in fatigue resistance. (Figure 3A)

Broken Surface Investigations of Covered RaCe Files

Plastic deformation is evident in the breaking section. In addition, the porous structure formed due to the coating is especially evident on the sharp edges. (Figure 3B)

Broken Surface Investigations of Covered TF Files

After coating, the surface has become quite rough. Traces of plastic deformation are evident on the sharp edges and are in the form of tearing. The crack started from the edge and progressed. The crack progression zone is smooth, the other is in the form of tearing. (Figure 3C)



Figure 3. Image of covered instruments after test procedures A: HeroShaper, B: Race, and C: TF

DISCUSSION

The first null hypothesis was rejected for 20° curved experimental setups. There were significant differences among the instruments in terms of the number of cycles to file breakage and time to fracture (p < 0.05). The first null hypothesis was partial rejected for 37° curved experimental setups. There were significant differences between HeroShaper and RaCe (p=0.000), and between TF and RaCe (p=0.000) in terms of the number of cycling to file breakage and time to fracture (p>0.05). However, there were no significant differences between HeroShaper and TF (p=0.121). The second null hypothesis was accepted for 20° curved experimental setups. There was no significant effect of the anodization process statistically among the groups. The second null hypothesis was rejected for 37° curved experimental setups. There was significant effect of the anodization process statistically among the groups. The third null hypothesis was rejected. As a result of the corrosion test, it was found that the anodization process had a positive effect on the HeroShaper file group. In RaCe and TF gruops, the anodization process reduced the corrosion resistance of the files.

A specification was determined by American Dental Association (ADA), which includes the physical properties, design features, and mechanical test procedures for endodontic files. The test procedure detailed in this specification No:28 is based on a static module. The tip of the instrument is fixed, then force is applied until breaking occurs, and the breaking resistances of the instrument against the torsional fatigue formed are calculated according to this module. Therefore, this is a reference to determine the resistance of Ni-Ti files to static loading. On the other hand, this specification is not sufficient and suitable for testing the dynamic properties of rotary system Ni-Ti files. In rotary systems, rotary files are used by rotating at a certain speed. Therefore, when testing rotary system files, it should be ensured that the ends rotate freely rather than being fixed.

In addition, since canal curvature is not considered when the test procedures are described in ADA 28 specification; metal fatigue in the instrument while working in curved root canals is not included in the evaluation. Whereas, the repetitive cyclic fatigue event caused by canal curvatures, is perhaps the most important factor in the sudden breakage of rotary

system files. (8,9). In our study, we investigated flexural fatigue that causes breaking due to the preparation of curved root canals.

In our study, we used a different modification of stainless-steel test setup, which is mostly preferred in recent studies (10-12). In this setup, artificial canals are prepared on a stainless-steel block, the file was tested without leaving its orbit during rotation. Thus, undesirable stresses in different directions and intensities due to the sliding of the file during rotation as in pin and tube assemblies were eliminated. Owing to the fiber glass covering the test setup, the file remained in the canal during the rotation, and the breaking moment of the file was clearly observed.

When the instruments are subjected to the fatigue test on the test setup, a lubricating gel is used to prevent friction between the file and the artificial canal and to prevent the heat and stress that may occur as a result of this friction. In some studies, synthetic oils, special oils, and especially in studies using the metal tubes as a test setup, glycerin has been used to prevent friction (10, 12,13,14). In our study, we used FileCare gel produced for Ni-Ti rotary systems for this purpose.

Many studies have reported that the curvature of the canal and the radius of the curvature are an important factor in metal fatigue, which is the biggest cause of instrument fractures during endodontic treatment (12,15,16). The results of these studies, the durability duration of the files decreased as the radius of curvature of the canal decreased. In addition, as the canal curvature increased, the breaking event occurred earlier. These different results obtained were also statistically significant (12,15,16). Our results support those of all these studies. When the results were evaluated, it was observed that the increase in

canal curvature significantly decreased the durability of the file for all file groups.

As the curvature of the root canal increases, the stress to which the file is exposed also increases. Each time the file goes round the canal the compressive and tensile forces increase parallel to the canal curvature. Increasing stresses and compressions create larger deformations in the structure of the file, reducing the lifetime of the file.

Studies on metal fatigue have investigated whether there is a difference between the different rotary system files in terms of lifespan (10,17,18).

In a study using Profile, K3, Hero, Mtwo, electropolishing RaCe, and non-electropolishing RaCe files, test parameters were the same for all file groups, canal curvature was determined as 45°, curvature radius as 5 mm, and rotation speed as 300 rpm ((Tripi et al. 2006). Only 25/.06 taper files from each file group were included in the study. Thus, variables such as file thickness and taper angle were kept constants for all experimental groups. When the results were evaluated, the highest value was recorded for Profile files, while the lowest value was recorded for non-electropolishing RaCe files. Since this difference was found to be statistically significant, it was stated that design differences such as cross-section geometry and surface properties are important factors affecting metal fatigue (17). Larsen et al. (10) investigated whether new generation Ni-Ti files have advantages over traditional files in terms of metal fatigue. For this purpose, TF and Profile GT X series files with different production characteristics were compared with traditional Profile and EndoSequence files. As in other studies, all test parameters were kept at the same values for all file groups. It was reported that Profile GT X files were more durable than all other file groups. 25/.04 TF files were found to be more resistant to breakage than EndoSequence files. However, it was reported that the difference between TF files and Profile files was not statistically significant.

In another study, the effects of different cross-sectional areas of K3, Profile, RaCe and TF files were investigated (19). In this study, 25/.06 files were tested with TF files at 500 rpm and all other files at 300 rpm. The times to breakage were recorded. Since different speeds were used, the number of cycles made by all files was calculated. TF files with the lowest cross-sectional area showed the highest breakage resistance, while K3 files with the highest cross-sectional area were reported to have the lowest breakage resistance. In the statistical evaluation, it was stated that there was a significant correlation between the cross-sectional area and file breakage due to metal fatigue (19).

In another study, Ni-Ti files produced by traditional methods and TF files produced by the twisted method were compared in terms of metal fatigue (Kim et al. 2010). For this purpose, 25/.06 TF, RaCe, Helix and Protaper F1 files were used in this study. The time to fracture was recorded. When the results were evaluated, TF files were reported to show higher resistance to metal fatigue than all other files (18).

In our study, results in 20° and 37° curved canals, the highest metal fatigue resistance was found in TF files. The difference among HeroShaper and RaCe files, and TF files was statistically significant (p<0.05). Our results support the results of the abovementioned studies using TF files. We think that the reason for the high fatigue resistance of TF files is that

they are produced with R-phase heat treatment technology.

In a study using RaCe files, RaCe files were reported to have higher resistance than Hero 642 files (17). Whereas, in our study looking at the results in both 20° and 37° curved canals, the lowest resistance to metal fatigue was recorded in RaCe files.

We attribute the reason for this difference between different file systems to the fact that the flexibility and resistance properties of the file differ from each other depending on the geometric shape properties, crosssectional area, and design differences.

Coating the metal surface with anodization is a method used in the field of implants in dentistry. It is aimed to facilitate osseointegration by coating the implant surface.

In this study, where we investigated whether this surface treatment would make a difference for Ni-Ti files, we recorded different results for different file groups.

In the 20° curved canal, the anodization process increased the wear time of RaCe and TF files while reducing the wear time for HeroShaper files. However, this increase, and decrease was not statistically significant (p>0.05).

In the 37 ° curved canal, the wear time of HeroShaper and RaCe files decreased while the wear time of TF files increased. These increases and decreases in the wear time of the files were found to be statistically significant (p<0.05).

Evaluating of the results of the corrosion test, the results showed that attention should be paid to anodization applications for files obtained from Ni-Ti alloy. The anodization process may show negative behavior especially depending on the geometry of the file. The complex geometry of the file prevented the

formation of a homogeneous and continuous oxide layer on the surface during the anodization process. This has a negative impact on corrosion resistance.

We think that the reason of different findings exhibited by different file groups may be the design differences of the files and/or the differences in the production phase, caused by the way of production.

The experimental procedures in this study were carried out at room temperature. However, instruments are exposed to body temperature in the clinical condition. The behavior of instruments changes in the different environmental conditions. Also, our experimental setup has only two-dimensional curve. But root canals have three-dimensional curve. The limitations of this study are such as above mentioned.

CONCLUSIONS

Within the limits of this study, increase in canal curvature reduces the life of the rotary instruments. Among the files used, the highest fatigue resistance was recorded in TF files at all curvature. While the anodization process does not change cyclic fatigue in all file groups in 20° curved canals, it reduced the wear life of RaCe and HeroShaper files and extended the wear life of TF files in canals with 37° curved canals.

Ethics Committee Approval: The Ethics Committee of graduate school of health sciences of Atatürk University, Erzurum, Turkey, approved (2011.3.1/7).

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