Effects of chelating agent (RC – Preparation) on surface characterization of profile nickel-titanium endodontic instruments

Dina Al-Sudani,* DDS, MsEdu
Saad Al-Shahrani,** DDS

هدفت من هذه الدراسة إلى دراسة تأثير مادة الـ(Rc–prep) على سطح الأدوات اللبية المصنوعة من مادة النيكل تيتانيوم. تم غمر الأدوات اللبية في مادة (Rc–prep) لأزمنة مختلفة وحتى مدة ساعة واحدة. تم تحليل العناصر السطحية لجميع الأدوات اللبية باستعمال المجهر الالكتروني ذو الطاقة الشعاعية التحليلية. أظهر التحليل السطحي أنه لايوجد تغير معتبر في كمية النيكل والتيتانيوم وأوكسيد النيكل في كل الأزمنة . لكن كان هناك زيادة إحصائية واضحة في تركيز ثاني أوكسيد التيتانيوم بعد خمسة دقائق , ونصف ساعة , وساعة من الغمر في الـ(Rc–prep) والنتيجة هي أن معالجة سطح المبارد المصنوعة من النيكل تيتانيوم بمادة الـ(Rc–prep) يؤثر على قبولها الحيوي.

The aim of the present study was to investigate the effect of a chelating agent, RC-prep, on the surface characterization of nickel-titanium endodontic instruments. The instruments were immersed in RC-prep for varying times up to 1 hour. The surface elements analysis of all instruments was carried out using electron microscope fitted energy dispersive x-ray (EDX) analyzer. Surface analysis revealed no significant change in the amount of Ni, Ti and NiO for all the time intervals. However, there was statistically significant increase in the concentration of TiO_2, after 5 minutes, ½ hour and 1 hour immersion times. In conclusion, the surface treatment of NiTi files with RC-prep may affect their biocompatibility.

INTRODUCTION

Root canal therapy, involves a variety of instruments used in conjunction with various intracanal medicaments such as sodium hypochlorite, H_2O_2 and RC-preparation. RC-prep is used for chemo-mechanical cleaning and shaping procedures. The chemical effects of these intracanal medicaments on endodontic files may hinder their performance as the chemical and electrochemical aggressiveness of these different solutions on the instrument themselves. [1,2]

After the pioneering research by Walia *et al.* [3], which introduced nickel-titanium (NiTi) to the endodontic profession, both nickel-titanium hand and rotary instruments have achieved widespread popularity. A major reason for their selection includes the properties of super-elasticity, shape memory, high corrosion resistance, and superior biocompatibility.[3] Despite this interest, a lot of work pertaining to the properties of NiTi instruments still has to be done.

Current endodontic practice necessitates that root canal instruments be repeatedly exposed to various sterilization procedures and irrigating solutions. RC-prep is generally accepted as the most effective chelating agent with prominent lubricant properties. It is used to enlarge root canal, remove the smear layer, and as a lubricant during instrumentation with most NiTi automated system. [4] Industrial testing has shown that EDTA is slightly corrosive to AISI 300 series stainless steel. Urea is corrosive to these materials at high temperature.[5] Several investigators have demonstrated the correlation between the surface change of chemical composition of NiTi files and some sterilization treatment.[6-17] The effects of these clinical practices on the surface of the NiTi endodontic files have not been determined.

The purpose of this study was to determine the effect of chelating agent (RC-prep) on the surface characterization of selected nickel titanium instruments.

Received 2 May 2005; Accepted 20 August 2005
*Assistant Professor
**Postgraduate Student
Department of Restorative Dental Science
College of Dentistry, King Saud University, Riyadh, KSA

Address reprint requests to :
Dr. Dina Al-Sudani
Endodontics Division
Department of Restorative Dental Sciences
King Saud University, College of Dentistry
P.O. Box 60169, Riyadh 11545, KSA
E-mail :drdina_2000@yahoo

METHODS AND MATERIALS

Fifty new Profile files were* used in the current investigation. The instruments were divided into 5 groups of 10 instruments each. Each group contained 2 instruments from each size (15, 20, 25, 30, and 35). Group I was used as a control and the other groups were exposed to RC-prep** for different time intervals. RC-prep used consisted mainly of 10% urea peroxide and 15% ethylene diamine tetracetic acid. The groups exposed to RC-prep were as follows:

Group I (Control): The unused files

Group II: The instruments were immersed in the RC-prep for 1 minute

Group III: The instruments were immersed in the RC-prep for 5 minutes

Group IV: The instruments were immersed in the RC-prep for ½ hour

Group V: The instruments were immersed in the RC-prep for 1 hour

Each file, after being immersed in RC-prep for the varying times at room temperature, was rinsed in distilled water and allowed to dry at room temperature on filter paper. Surface elements analysis (spot analysis) of all the instruments was carried out using electron microscope fitted energy dispersive x-ray (EDX) analyzer (Link Oxford Analytic System) under 200X magnification (Fig. 1). The mean value and standard deviation for each element on the surface was recorded and *t*-test was used for comparison among the groups.

*Maillefer Instruments SA, Bailegues, Switzerland
**Primer Dental Pruducts Co., King of Prussia, PA

RESULTS

Mean and standard deviation for each element (Ni, Ti, NiO and TiO_2) which were measured on the files surface before and after exposure to RC-prep are shown in Tables 1 and 2. There was no statistically significant difference ($P>0.05$) of Ni or Ti concentration for all time intervals Table 1. Moreover, as shown in Table 2 there was no significant change ($P>0.05$) in the concentration of the nickel oxide (NiO) for all time intervals. There was no significant change ($P>0.05$) in the concentration of the titanium oxide (TiO_2) after immersion for 1 minute. However, there was statistically significant increase ($P<0.05$) in the concentration of the TiO_2 after exposure for 5 minutes, ½ hour and 1 hour (Table 2 and Fig. 2).

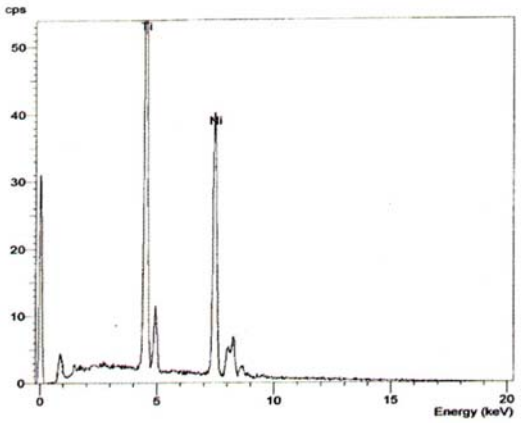

Fig. 1. Surface elements analysis using energy dispersive x-ray (EDX).

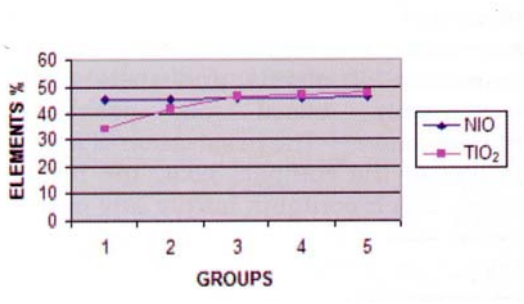

Fig. 2. The concentration (%) of NiO and TiO2 after exposure to RC-Prep for varying duration.

Table 1. The concentration (%) of Nickel (Ni) and Titanium (Ti) before and after exposure to RC-prep for varying duration.

Group	Conc. of Ni	Conc. of Ti
	Mean ± SD	Mean ± SD
I (Control)	2 ± 8.6.45	45.0 ± 6.1
II (1 min)	42.8 ± 4.0	42.9 ± 3.4
III (5 mins)	41.6 ± 6.4	44.3 ± 4.7
IV (½ hr)	43.2 ± 7.6	45.6 ± 6.5
V (1 hr)	41.8 ± 4.2	45.0 ± 3.4
	P>0.05	*P*>0.05

Fig 2. The concentration (%) of NIO and TIO_2 after exposure to RC Prep for varying duration

Group	Conc. of NiO	Conc. of TiO_2
	Mean ± SD	Mean ± SD
I (Control)	45.0 ± 11.8	33.8 ± 11.5
II (1 min)	45.5 ± 2.6	41.6 ± 14.8
III (5 mins)	45.6 ± 1.8	46.3 ± 2.8*
IV (½ hr)	46.0 ± 1.5	46.9 ± 1.9*
V (1 hr)	46.4 ± 0.5	*48.0 ± 0.5**
	P>0.05	*P*>0.05 **P*<0.05

DISCUSSION

The surface elements of NiTi files were studied after they were exposed to RC-prep, which is generally accepted as the most effective chelating agent with prominent lubricant properties in endodontic treatment.[4] The results indicated that the surface of NiTi files consisted mainly of TiO_2, NiO, and metallic Ni, while nickel-titanium compounds constitutes the inner layer. The finding of the present study parallels the results of some other investigators. [7-9]

NiTi files have stable oxide layer on their surfaces that helps to render them corrosion resistant.[13] The corrosion resistance of metals and metal alloys is mainly based on passivation phenomenon.[13] The passivation of a metal is due to the compact coat, the passive layer, which contains hardly any original metal, but forms a metal oxide layer, "skin," on metal. This oxide layer may be amorphic or crystalline. The passive oxide layer varies in thickness depending on the preparation methods and could be enhanced by modifying the thickness, topography and chemical composition of this oxide layer by different surface treatment.[7, 11-13] In our present study, the exposures of NiTi file to RC-prep which contains mainly EDTA and urea peroxide resulted in the increase of the oxide layer. This finding was in agreement with Villermaux *et al.*[12] They showed that nitric acid passivation is recommended for surface treatment of medical devices to remove plastically deformed native oxide layers and replace them by more uniform ones. This improved oxide layer has been shown to be very protective and promising for improving the corrosion resistance and biocompatibility of NiTi, which was reported by many authors.[12,13,15] Several *in vitro* evaluations of the corrosion properties of the alloy have been preformed.[13,16,17] Ni-Ti alloy derives its good corrosion behavior from a homogeneously covered surface of Ni-Ti alloy in the presence of oxygen.[11,14] Furthermore, it was found that the uniformity of the oxide layer rather than its thickness and composition, seemed to be the predominant factor to explain the improvement of corrosion resistant.[7,11,15-17] Sohmura[13] also showed that this layer is able to sustain large deformations induced by the shape memory effect.

Corrosion reactions susceptible to occur with clinical use of NiTi are numerous and can lead to different rates of ionic release ranging from low rates during passive dissolution to high rate when breakdown of the protective oxide film occurs.[16,17] The current study demonstrated that the surface treatment performed on NiTi had modified the amount of the protective oxide layer on their surface which promoted their good biocompatibility and their excellent resistance to corrosion processes. According to several researchers, the

corrosion behavior is directly related to the surface property.[16,17] Moreover, this result could be attributed to the effect of RC-prep treatment in addition to the presence of oxygen which is known to cover the titanium alloy spontaneously by TiO_2 layer.[11,14,15,17] However, it was indicated by previous studies[7,11,15-17] that the uniformity of the oxide layer, rather than its thickness and composition, seemed to be the predominant factor to explain the improvement of corrosion resistance. Further investigation has to be done to study the uniformity of this oxide layer. From the current findings, it was concluded that the RC-prep surface treatment performed on NiTi had enhanced the protective oxide layer which was found in previous studies to be the predominant factor to explain the improvement of corrosion resistant of Ni-Ti instruments.

CONCLUSION

The surface treatment of NiTi files with RC-prep may affect their biocompatibility.

ACKNOWLEDGEMENT

The author would like to express her great appreciation to Prof. S. Kheir for her invaluable insight and discussions regarding this paper.

REFERENCES

1. Gutierrez JH, Gigoux C, Sanhueza I. Physical and chemical deterioration of endodontic reamers during mechanical preparation. Oral Surg 1969; 28: 395-403.
2. Eichner MA, Schoen DM, Goldman M, Kronman JH. Effect of protein and sodium hypochlorite on endodontic instruments. J Endod 1976; 2: 335-338.
3. Walia H, Brantley WA, Gerstein H. An initial investigation of the bending and torsional properties of Nitinol root canal files. J Endod 1988; 14:346-351.
4. Rudle C. Cleaning and shaping the root canal system. In: Cohen S, Burns R. Pathways of the Pulp, 8th ed. St. Louis: CV Mosby Inc., 2002 pp. 231-289.
5. Neal R, Craig R, Powers J. Effect of sterilization and irrigation on the cutting ability of stainless steel files. J Endod 1983; 9: 93-96.
6. Shabalovskaya SA, Andereeg J. Surface spectroscopic characterization of Ni-Ti equiatomic shape memory alloys for implants. Jvag Sci Technol 1995; 13; 2624-2632.
7. Shabalovskaya SA, Cunnick J, Anderegg J, Harmon B, Sachdeva R. Preliminary data on *in vitro* study of proliferative rat spleen cell response to Ni-Ti surface characterized using ESCA analysis. In: Proceedings of the International Conference on Shape Memory and Superelasticity. Pacific Grove, CA 1997, pp. 209-214.
8. Hanawa T, Oda M. Calcium phosphates naturally formed on titanium in electrolyte solution. Biomaterials 1991; 12:767-774.
9. Oshida Y, Schder RCL, Miyazaki S. Microanalytical characterization and surface modification of Ni-Ti orthodontic arch wires. Biomed Mater Eng 1992; 2:51-69.
10. Kruger J. Fundamental aspects of the corrosion of metallic implants: In Rubin LR (Ed) Biomaterials in reconstructive surgery. St. Louis: CV Mosby pp. 145-157.
11. Trçpanier C, Tabrizian M, Yahia L, Bilodeau L and Piron L. Effect of modification of oxide layer on NiTi stent corrosion resistance. J Biomed Mater Res 1998; 43: 433-446.
12. Villermaux F, Tabrizian M, Yahia L'H, Meunier, M, Piron DL. Excimer laser treatment of Ni-Ti shape memory alloy biomaterials. Appl Surface Sci 1997; 110:62-66.
13. Sohmura T. Improvement in corrosion resistance in Ti-Ni shape memory alloy for implant by oxide film coating. World Biomaterial Congress Proceeding, Kyoto, Japan; 1988:574.
14. Annual Book of ASTM Standards, Vol. 13 No. 01. Philadelphia, PA: ASTM; 1996: Section 13.

15. Espinos JP, Fernandez A, Gonzales-Elipe AR. Oxidation and diffusion processes in nickel-titanium oxide systems. Surface Sci 1993; 295:402-410.
16. Theierry B, Tabrizian M, Savadogo O, Yahia L. Effect of sterilization processes on NiTi alloy: Surface characterization. J Biomed Mater Res 1999; 48: 165-171.
17. Thierry B, Tabrizian M, Trepanier C, Savadogo O, Yahia L. Effect of surface treatment and sterilization processes on the corrosion behavior of Ni-Ti shape memory alloy. J Biomed Mater Res 2000; 51:685-693.