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Website: www.jorthodsci.org DOI: 10.4103/jos.JOS 55 20

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Submitted: 25-Aug-2020 Revised: 14-Feb-2021 Accepted: 05-Mar-2021 Published: 04-Aug-2021

The effect of fluoride gel on tensile properties, surface morphology and chemical composition of two types of orthodontic wires (an *in-vitro* study)

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Abstract:

OBJECTIVE: Stainless steel and nickel-titanium wires are commonly used in fixed orthodontic appliances. Orthodontists often prescribe fluoride gel to avoid demineralization of teeth. This study investigated the effect of acidulated phosphate fluoride gel on the tensile properties, surface morphology and surface chemical composition of stainless steel and nickel-titanium wires.

METHODS: Forty samples of stainless steel and nickel-titanium wires were examined, twenty for each type. Each wire type was divided into four subgroups. The first subgroup was not immersed in fluoride gel and considered as control, while the other three subgroups were immersed in 10 mL of fluoride gel for different periods of time. Then, the wires were removed from the gel, rinsed in a distilled water and left to dry. The ultimate tensile force of each wire was measured using a tensile testing machine. Scanning electron microscope (SEM) and energy-dispersive X-ray spectroscopy (EDX) were used to examine the control and 24 h immersed subgroups.

RESULTS: One-way analysis of variance ANOVA showed that there were no significant differences in the ultimate tensile forces between control and fluoride-treated subgroups (P = 0.172 for SS wires and P = 0.672 for NiTi wires). However, changes in the surface morphology and elemental composition of wires were shown by the SEM and EDX.

CONCLUSIONS: Although immersion of stainless steel and nickel-titanium wires in the fluoride gel did not affect their tensile properties, however, surface deterioration was evident. So, further investigations are recommended to study the effect of these changes on the oral health of patients.

Keywords:

Chemical composition of orthodontic wires, fluoride gel, orthodontic wires, surface morphology, tensile force

Introduction

Orthodontic wires are usually used to exert light constant forces on teeth through brackets and tubes in order to initiate teeth movement.^[1] Many types of arch-wires were manufactured in the process of finding the best material to achieve the required goals of orthodontic treatment. Stainless steel (SS) alloy is

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commonly used in orthodontic wires because of its strength, resilience, compatibility, and formability.^[2,3] The most SS alloy regularly used for orthodontic materials is the American Iron and Steel Institute type 304, which contains 8%–10% nickel and 18%–20% chromium. Nickel reinforces austenitic phase of SS while chromium increases its resistance to corrosion through forming a passive oxide layer Cr_2O_3 .^[4-6] Oxide layer prevents oxygen from diffusion into that underlying alloy. In addition, nickel competes with

How to cite this article: Taqa AA, Al-Hafidh NN, Al-Abbood MT. The effect of fluoride gel on tensile properties, surface morphology and chemical composition of two types of orthodontic wires (an *in-vitro* study). J Orthodont Sci 2021;10:14.

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chromium to form salts made more chromium available to form the oxide layer and obtain a stable austenitic structure.^[4] Nickel-titanium (NiTi) were used in Orthodontics since 1971. Nickel-titanium wires (55% nickel and 45% titanium) have a wide activation and deflection range and deliver low forces because of its high elasticity. Hence, it is widely used through the initial stage of orthodontic treatment.^[6-8] On the other hand, SS wires are used in the later stages of treatment because they deliver larger forces.^[2,3,7,9] Both types of wires showed more susceptible to damage due to chemical and physical factors when they subjected to oral environment for a long time. Orthodontic patients are more susceptible to have white spot lesions on their teeth due to stagnation of food inside the orthodontic appliances.^[10,11] Therefore, orthodontists prescribe fluoride-containing products to prevent the white spot lesions. Fluoride plays an important role in the remineralization of teeth.^[12,13] Previous studies were conducted to investigate the effect of fluoride-containing products on the surface characteristics and mechanical properties of orthodontic arch-wires.^[14-17] Some of these studies reported changes in unloading mechanical properties.^[15,16] Conversely, other studies did not find a significant decrease in the unloading and mechanical properties of arch-wires after immersion in fluoride-containing products.[18-21] Abbasy et al.^[22] found that the Acidulated Phosphate Fluoride (APF) gel caused an increase in the friction resistance of NiTi wires. However, they did not address to the effect of APF gel on the tensile force and surface chemical composition of the wires. Since these wires are subjected to oral environment, any weakness due to the APF gel made them more susceptible to deterioration and damage through orthodontic treatment. Moreover, the biodegradation of wires due to chemical factors may release elements from the metallic alloy of wires. In turn, these elements may cause allergic reactions and potential cytotoxic effects that may jeopardize the health of patients.^[23-25] Thus, extra information regarding the effect of APF gel on the surface characteristics of orthodontic wires is important. Furthermore, advance imaging techniques such as Energy Dispersive X-Ray Spectroscopy (EDX) which is the most standard and consistent method in the field of analytical electron microscopy can provide more detailed information about the effect of APF gel on orthodontic wires.^[26,27] The main aim of this study was to investigate the effect of APF gel on the tensile force of SS and NiTi orthodontic wires. Besides, it also aims to observe the surface characteristics and elemental composition changes of commercial APF gel on these wires using SEM and EDX. The null hypothesis states that there is no effect of fluoride gel on the tensile force and surface characteristics of the SS and NiTi orthodontic wires.

Materials and Method

Study samples

This research was conducted according to the principles of the Declaration of Helsinki. Samples were divided into two groups: SS and NiTi wires, each group consists of four subgroups. Each subgroup consists of five wires; each wire is (0.016 inch) 0.40 mm in diameter and 80 mm in length. The calculation of sample size (*n*) was done according to the following formula, $n = (z\sigma/D)^2$, where (*z*) is a constant has a value of 1.282 for a confidence level of power 80%, (σ) is the standard deviation and equals to (0.51) from primary data and precision (D = 0.3). Since n = 4.749, therefore, five samples for each subgroup were adopted.

Materials used in this study were SS remanium[®] stangendran (Dentarum, Ispringen, Germany) and NiTi straight length wires (G&H Wire Company, Early wood Drive Franklin, IN 46131, USA). Table 1 illustrates the chemical composition of SS and NiTi wires. The fluoride gel that examined was TOPEX acidulated phosphate of 1.23% fluoride ion and pH 3.5 manufactured by (Sultan Healthcare Inc., 85 West Forest Avenue, Englewood, USA).

Subgroup 1 represents the SS straight wires which were not immersed in the fluoride gel and considered as control. Subgroups 2, 3, and 4 are the SS wires immersed in the 10 mL APF gel for 5 min, 1 h and 24 h, respectively. Subgroup 5 is the NiTi wires those were not immersed in the fluoride gel and considered as control, subgroups 6, 7, and 8 are the NiTi wires immersed in 10 mL APF gel for 5 min, 1 h, and 24 h, respectively. Then, all of the immersed subgroups were removed from the APF gel and rinsed with distilled water then they were left to dry.

The outer surfaces of SS and NiTi wires were observed by SEM (FEI Company, Netherlands, Inspects 50 mode) before and after immersing in the APF gel for 24 h. The elemental composition of SS and NiTi wires was

Table 1: Ch	emical com	position of SS and Ni	Ti wires
Chemical composition of SS wire (remanium)		Chemical composition of NiTi wire	
Element	(Weight %)	Element	(Weight %)
DIN, AISI	1.431	Nickel	54.5-57.0
Carbon	0.05-0.15	Carbon	<0.050
Silicon	≤2.0	Cobalt	<0.050
Manganese	≤2.0	Copper	<0.010
Chromium	16.0-19.0	Chromium	<0.010
Molybdenum	≤8.0	Hydrogen	<0.005
Nickel	6.0-9.5	Iron	<0.050
Phosphorus	≤0.045	Niobium	< 0.025
Sulfur	≤0.015	Nitrogen plus Oxygen	<0.050
Nitrogen	≤0.11	Any single trace element	<0.1
Iron	Residue	Total Trace Element	<0.25
		Titanium	Balance

measured by EDX (XFlash_6l10 model, Bruker Company, Germany) before and after immersing in APF gel for 24 h. Each sample was examined by two methods of EDX, standard and oxide methods.

Tensile test

Each wire was fixed with two screws at each end [Figure 1a]. The screws are mounted into plastic cylinders filled with self-cured acrylic [Figure 1b]. The wire span between cylinders was unified to be 40 mm. This method was adopted to prevent any slippage of wire and enable sample grasping by the cross-head of the testing machine. The tensile tests of samples were conducted on a fully computerized versatile electronic testing machine (Sans Testing Machine Co. Ltd., Shenzhen, China) [Figure 2a]. The cross-head speed of the machine was 3 mm/min. The statistical software for social sciences (SPSS version 18.0, SPSS Inc., Chicago, Illinois, USA) was used to perform the statistical analyses. The analyses included descriptive statistics for each subgroup and one-way ANOVA multi-variance analysis. Besides, the analyses were performed to assess which subgroups immersed in APF gel are significantly different from the control subgroups. It is important to mention that the length of the wire was measured again after test and it was found to have the same length as before the test of 40 mm, which meant that no slippage occurred. Figure 2b shows a wire sample after test.

Results

The mean values of the ultimate tensile force (UTF) of different subgroups are shown in Table 2. No significant differences were observed in the UTF values at (P < 0.05) among control subgroups and subgroups immersed in the APF gel for both SS and NiTi wires (P = 0.172 for SS wires and P = 0.672 for NiTi wires).

SEM images of control SS wires and SS wires immersed in the APF gel for 24 h with different magnification powers (\times 500, \times 1000, and \times 3000) are shown in Figure 3a

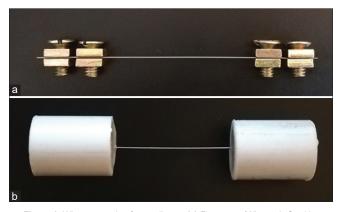


Figure 1: Wire preparation for tensile test (a) First stage (Wire ends fixed by screws) (b) Second stage (Wire ends fixed by acrylic)

and b. SEM images showed that immersed SS wires had more pitting and scratches lines on their surfaces compared to control SS wires. For NiTi wires, SEM images showed that wires immersed in the APF gel for 24 h compared to control wires had rougher and more obvious deterioration surfaces with pitting and islands-liked areas as shown in Figure 3c and d.

Figure 4a and c present the elemental composition of outer surface of control SS wire. While Figure 4b and d show elemental composition of outer surface of SS wires which immersed in APF gel in 24 h. The results of EDX revealed a disappearance of carbon and decrease in chromium after immersion of SS wires in the APF gel as shown in Table 3. On the other hand, Figure 4e and g illustrated the elemental composition of outer surface of control NiTi in both oxide and standard methods. While Figure 4f and h described the elemental composition of NiTi wires that immersed in APF gel for 24 h. Table 3 discloses an appearance of Ni, Al, Ta, Zr, and C trace elements in the surface of immersed NiTi wires.

Discussion

This study examined both SS and NiTi wires because they are used at different stages of orthodontic

Table	2:	Mean±standard	deviation	of	ultimate	tensile
force	(N)					

Type of wire	Mean value of ultimate tensile force (UTF) (Newton)	Standard deviation (SD)
SS (control subgroup)	303.5	7.7
SS + Fluoride (5 min)	308.2	10.1
SS + Fluoride (1 h)	319	0.6
SS + Fluoride (24 h)	310.8	8.6
NiTi (control subgroup)	182.6	4.3
NiTi + Fluoride (5 min)	183.5	1.1
NiTi + Fluoride (1 h)	184.4	1.0
NiTi + Fluoride (24 h)	180.8	5.4

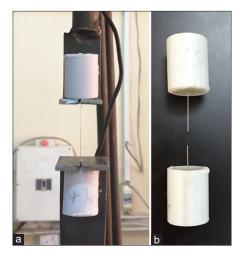


Figure 2: Tensile test of a sample (a) Before tensile test (b) After tensile test

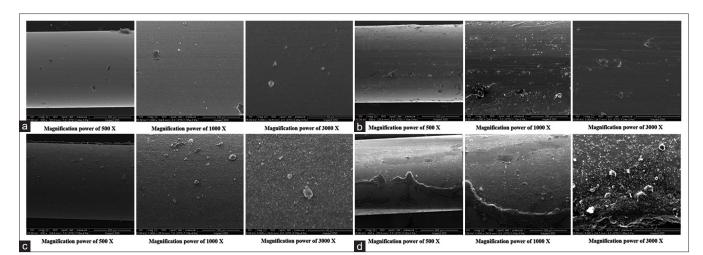


Figure 3: SEM images of SS and NiTi wires (a) Control SS wire (b) SS wire immersed in APF gel for 24 h (c) Control NiTi wire (d) NiTi wire immersed in APF gel for 24 h

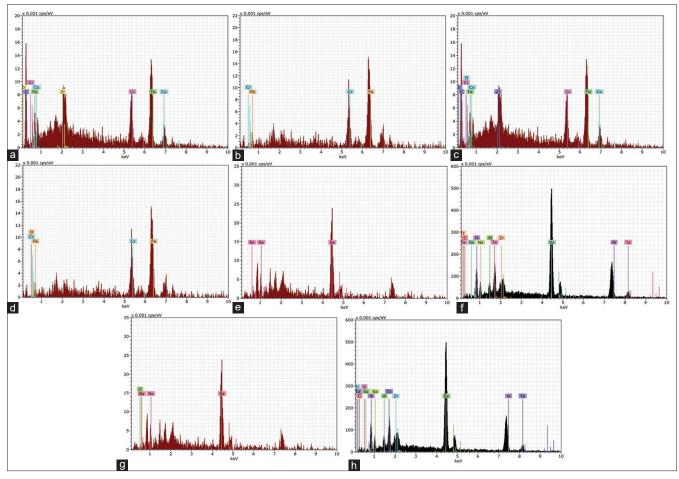


Figure 4: EDX of SS and NiTi wires (a) Standard method of control SS wire (b) Standard method of immersed SS wire in APF gel for 24 h (c) Oxide method of control SS wire (d) Oxide method of immersed SS wire in APF gel for 24 h (e) Standard method of control NiTi wire (f) Standard method of immersed NiTi wire in APF gel for 24 h (g) Oxide method of control NiTi wire (h) Oxide method of immersed NiTi wire in APF gel for 24 h (g)

treatment and they remain in patient mouth for a long time. As a result, they are subjected to oral environment including APF gel prescribed by orthodontists. Based on the results of this study, the null hypothesis regarding the effect of APF gel on tensile force was accepted. Yet, the findings of SEM and EDX revealed surface

deterioration and changes elemental composition of SS and NiTi wires.

The durations of fluoride application that adopted in this study were 5 min, 1 h, and 24 h. The 5 min duration was chosen to simulate the clinical instructions whereas

Table 3: E	EDX measui	rements of S	SS and	NiTi wires

Element	Control SS (Weight %)	SS + Fluoride 24 h (Weight %)
SS wires (Standard method)		
Iron 26-series	16.856	41.2414641
Chromium 24-series	5.0235	2.72711750
Cobalt 27-series	6.3091	
Zirconium 40-series	3.5650	
Carbon 6-series	15.193	
SS wires (Oxide method)		
Iron	0.3514278	0.4059
Chromium	0.1056689	0.0287
Cobalt	0.1317725	
Zirconium	0.0541887	
Oxygen	0.1684593	0.1295
Carbon	0	0
FeO	0.452107035	0.52216
Cr ₂ O ₃	0.544411560	0.04201
ZrÔ	0.073196581	
NiTi wires (Standard method)		
Barium	78.786	89.66
Sodium	1.3946	2.81
Nickel		34.09
Tantalum		24.49
Zirconium		5.67
Aluminum		1.48
Carbon		1.12
NiTi wires (Oxide method)		
Barium	0.924069995	81.725
Sodium	0.015882240	2.4672
Oxygen	0.0113184067	21.725
Nickel		30.846
Tantalum		22.127
Zirconium		5.1053
Aluminum		1.2881
BaO		91.2466
Na2O		3.32575
Al ₂ O ₃		2.43388
ZrO ₂		6.89604
NiO		39.2540

the 1 h duration was selected based on an *in vitro* study presented by Abbasy *et al.*^[22] who stated that immersion of wires in fluoride gel for one h equalizes 21 days of daily brushing with fluoridated toothpaste. Unlike previous studies, the 24 h duration was adopted in this study as a worse case to investigate the long-lasting effect of APF gel on wire materials.

Toms^[4] stated that the UTF value reflects the generalized and localized corrosion effects on orthodontic wires, therefore, this study adopted the UTF value for each test. The study showed that there is no significant difference in the UTF values among subgroups of control and APF gel immersed for 5 min, 1 h, and 24 h for each type of wires. A direct comparison of obtained results with other previous studies was not possible because of using different methodologies. However, a recent study reported that there is no significant difference in the tensile strength of SS wires after exposure to fluoridated mouthwash.^[20] On the other hand, Walker *et al.*^[16] stated that SS wires showed a statistically significant decrease in unloading mechanical properties after exposure to either neutral or acidulated fluoride gel. They also stated that this decrease is not great enough to be clinically significant. In our study, SEM images showed that surface changes occurred after immersing of SS wires in the APF gel for 24 h as an increase of pitting on the wire surface [Figure 3a and b]. This came in accordance with the results of other studies.^[16,17]

Corrosion resistance of wires alloys depends on their passive surface layer.^[12] This layer protects alloys of SS and NiTi from corrosion and once it deteriorates, the alloys will be prone to corrosion.^[6] However, the key factor of corrosion resistance of orthodontic wires is the surface chemical composition.^[19]

In our study, EDX showed a presence of iron, chromium, cobalt, zirconium, and carbon on the surface of control SS [Table 3]. The presence of carbon probably indicates that there is a carbonated organic adhesive or binding compound in the composition of SS alloy. Carbon adds strength to SS alloy and increases its corrosion resistance.^[4] Yet, the SS wire that immersed in APF gel for 24 h, carbon disappeared, chromium declined and iron increased due to liberation of iron after chemical disruption of the oxide protective layer by fluoride ion effect and hydrofluoric acid of APF gel [Figure 4a-d and Table 3].

This study also showed that there is no significant difference in the UTF values of NiTi wires after immersion in the APF gel for 24 h. This result coincides with a study conducted by Perinetti et al.[19] who documented significant surface corrosion without significant degradation in the tensile mechanical properties. Furthermore, Srivastava et al.[18] found similar results despite measuring the mechanical deflection properties using the three-point bending test. They concluded that there is no significant reduction in the mechanical properties of SS and NiTi wires after immersion in 0.05% Phosflur and 0.2% neutral NaF mouth rinses. Another in-vivo study reported that commercial fluoride rinse and fluoride gel used once a day for a whole month did not affect the mechanical loading properties of NiTi wires.^[21] Unlike Srivastava et al.^[18] and Perinetti et al.,^[19] this study used EDX to illustrated the deterioration and changes in the elemental composition of wire surfaces.

The EDX standard method of control NiTi showed a presence of barium and sodium ions while EDX standard method of immersed NiTi wires showed an increase of barium and sodium ions and appearance of other elements wires such as: nickel, titanium, zirconium, aluminum, and carbon [Table 3]. This result could be due to dissociation of protective oxide layer. In addition, the EDX oxide method of NiTi wires immersed in APF gel for 24 h showed further compounds of BaO, NiO, $ZrO_{2'}$ Na₂O and Al₂O₃ [Figure 4h].

This liberation of trace elements pointed to change of the elemental composition of NiTi wire after immersion in APF gel. This finding might indicate that even when surface corrosion is not big enough to destruct the metal component of SS and NiTi wires; it obviously changes their surface chemical composition. Nickel and chromium have potential for producing allergic, toxic, or carcinogenic effects.^[28] In summary, the effect of such trace elements and corrosion products on patients' oral health must be considered.

Although the effect of APF gel on the surface deterioration of tested wires was noticeable in this study, many interrelated variables such as acidity of certain food and drink, bacterial activity, salivary flow rate, pH and buffer capacity of saliva could affect the influence of APF gel on wires in the oral environment.^[29] However, the inclusion of these variables in this study was unfeasible because of the difficulty in simulating them accurately in an in-vitro study. In addition, the standardization of these variables in patient mouth is quite difficult.

Conclusion

Immersion of SS remanium[®] and NiTi orthodontic wires in the APF gel for 5 min, 1 h and 24 h did not significantly affect the UTF values of wires. However, serious surface topography deterioration of both SS remanium[®] and NiTi wires was noticed by SEM. In addition, the changes in elemental composition of SS and NiTi wires were evident by EDX. To conclude, orthodontists should be aware of any cumulative effects of APF gel on wires and thus the patients' oral health.

Acknowledgements

The authors would like to thank Dr. Aymen A. Ahmed, a lecturer at the Department of Biophysics at the University of Mosul for his valuable review of this research.

Financial support and sponsorship Nil.

Conflicts of interest

There are no conflicts of interest.

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