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EFFECT OF REMINERALIZING AGENTS ON THE FLEXURAL MODULUS OF ELASTICITY OF NICKEL TITANIUM AND COPPER NICKEL TITANIUM WIRES

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ABSTRACT

Objective: To determine the effect of acidulated phosphate fluoride "APF" and casein phosphoprotein amorphous calcium phosphate "MI paste" on the flexural modulus of two super elastic orthodontic wires.

Material and Methods: Preformed round and rectangular NiTi and Cu NiTi were immersed in either APF, MI paste or artificial saliva (control) for 4 minutes. The wires were then subjected to a 3-point bend test in accordance with the American National Standard/American Dental Association Specification No. 32 (2006)

Results: NiTi wires were significantly affected by the prophylactic agents' application with a more substantial effect for those treated with APF. The effects were less in magnitude however still significant for the tested Cu NiTi wires.

Conclusion: Mechanical properties of NiTi wires are significantly affected by the application of remineralizing agents. Clinically it would preferable to use Cu Niti wires if it is predicted that the patient will need a prophylactic agent.

INTRODUCTION

The orthodontic arch wire is one of the main parts of the multibracket appliance. It is designed to move teeth from malocclusion to a preferred dental occlusion through mechanical interaction with the bracket slots. Orthodontic treatment mostly constitutes of different phases with special requirements of force delivery and mechanotherapy during each phase.^{1,2} Generally, it is vital that wires used should generate a continuous light force within each phase of treatment ³. At the same time the orthodontist's goal is to satisfactorily accomplish treatment in the least amount of time with minimum patient discomfort, while avoiding complications by maintaining healthy teeth and supporting structures.⁴

Manufacturers attempt to produce wires with specific physical and mechanical properties to

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satisfy orthodontic requirements yet these properties are liable to change under various manipulations and conditions⁵. One such condition affecting the mechanical properties of arch wires is the use of dental re-mineralizing agents which is sometimes mandatory to preserve the dental structure⁶.

These agents are also used to enhance the patients' oral hygiene and as a prophylaxis to reduce the incidence of the white spot lesions commonly associated with orthodontic fixed appliances^{7,8,9}. Products containing fluorides and casein phosphoprotein are the mainly prescribed prophylactic agents used for such purposes⁷⁻⁹. Different protocols including professional in-office as well as home use applications are advocated throughout the orthodontic treatment period. Therefore, it is important to understand their effect on the various components of the orthodontic fixed appliances. The unique properties of superelastisty and shape memory allow titanium-based wires to deliver a constant force over a wide range of activation making them ideal for orthodontic tooth movement^{4,13}. Accurate knowledge of the mechanical properties of these wire is essential in their utilization to deliver ideal forces for tooth movement14,15.

Titanium and titanium based alloys are known for their excellent corrosion resistance attributed to their protective oxide^{6,13}. However previous studies have recorded discolorations, surface changes as well as changes in mechanical properties of superelastic titanium based wires in response to fluoride applications^{5,6}. Therefore, this study was conducted to test and compare the effects of acidulated phosphate fluoride "APF" and casein phosphoprotein amorphous calcium phosphate "MI paste" on the flexural modulus of NiTi and Cu NiTi wires as well as any possible influence wire cross section might have on that interaction.

MATERIAL AND METHODS

Preformed Ormco pseudo nickel titanium (NiTi) round and rectangular wires as well as thermoplastic copper nickel titanium wires (Cu NiTi) were chosen for their superelastic properties and popularity.

	Material	Cross-section in Inches	Number of Wires
Group 1	Niti	0.016	50
Group 2	Niti	17x25	50
Group 3	Cu-Niti	0.016	50
Group 4	Cu-Niti	17x25	50

TABLE (2) Number of wire samples distributed according to; wire alloy and wire crosssection

The fluoride agent used was 1.23% APF, Sorbet ,Dental Resources, a division of Key Stone Industry, Myerstown, Pennsylvania, USA and the casein phosphoprotein was MI Paste, GC International, Itabashi-Ku, Tokyo, Japan. (table 1)

TABLE (1) Prophylactic agents; composition and manufacturing company

Prophylactic Agent	Composition	Manufacturer
APF "Sorbet"™	Acidulated phosphate fluoride. Flouride ion 1.23% available from 2.09% sodium fluoride and hydrofluoric acid	Dental Resources, A division of Key Stone Industry, Myerstown, Pennsylvania, USA
"MI Paste" тм	Casine phosphoprotein amorphous calcium phosphate	GC International, Itabashi-Ku, Tokyo, Japan

Each wire specimen was cut from the straight portion of preformed arch wires with a round crosssectional dimension of 0.016 inch and a rectangular cross- sectional dimension of 0.017x0.025 inch and were cut to the length of 20 mm.

The wire specimens were divided into four equal groups of 50 according to the wire alloy and cross section. (Table 2)

Wire treatments

From each sub-group 10 wire specimens were allocated to be used as control samples. These wires were immersed in separate vials containing artificial saliva for 5 minutes prior to their testing. The remaining wires from each sub-group were randomly selected to be treated with either; APF or MI Paste rendering 20 wires for each treatment. The treatment consisted of a four-minute application of the prophylactic agent (total emersion of the wire in the agent in a glass bowl). After that, 10 wires of each treatment subgroup (no. of wires per sub group 20) were randomly allocated to be tested immediately after prophylactic agent application. Those wires were treated individually (one at a time) with the prophylactic agent then removed with tweezers and washed in a bowl with artificial saliva (the wires of each treatment were washed in a separate artificial saliva bowl) and tested immediately (T0). The prophylactic agent application and washing procedures were repeated for the remaining 10 wires in each subgroup allocated for delayed testing. This was followed by their storage in separate test tubes containing artificial saliva for 3 weeks. These subgroups were then tested at (T1).

Testing procedure

In our study, all specimens were subjected to a 3-point bending test as described by Miura et al²³. Mechanical testing was also based on the current American National Standard/American Dental Association Specification No. 32 for Orthodontic Wires 2006. ⁵

As indicated in the specification, the wire specimens were submerged and tested in a heated distilled H_2O bath (37±1 °C) to simulate the aqueous oral environment. The temperature of the water bath was maintained via a water heater and a thermocouple thermometer.

The mechanical testing was performed on a computer controlled universal testing machine (Lloyd Instruments LR 5K Serial No 014441; Load cell 5 KN) and the data was collected using Nexygen version 4.5. The testing procedures were performed at the Dental Biomaterial Department, Faculty of Dentistry, Ain Shams University.

During the testing procedure, the wire was supported by a jig, the jig span would determine the length of the wire that was subjected to the bending test. For this test and its clinical applications, the testing procedures were customized for each wire alloy. According to a pilot testing, Titanium based alloys were tested on a 12mm custom made jig span. (Figure 1).

Compressive force was applied at a crosshead speed of 1 mm/min by means of a striker (steel rod with a bi-beveled chisel end) placed midway between the two poles of the jig. Each specimen was loaded to a deflection of 3.1 mm and then unloaded at the same crosshead speed until zero deflection was reached. Load in newtons (N) and deflection in



Fig. (1) Custom made testing jigs

millimeters (mm) were recorded for each specimen with the Nexygen computer software program. Load deflection curves were the generated via MS Excel (2010).

The engineering beam theory was used to calculate the flexural modulus of elasticity (E).E for the loading slopes of round cross section specimens was calculated using the following equation:

 $E = 4L^{3}m/3\pi D^{4}$ (GPa).

E for the loading slopes for rectangular specimen was calculated using the following equation⁹⁵:

 $E = L^3 m/4bd^3 (GPa).$

P = load at the apparent yield point (N), L = support span (mm), b = width of specimen (mm), d = depth of specimen (mm) and m = slope of the straight-line portion of the loading deflection curve (N/mm of deflection).

Statistical Method

The Generalized Linear Model was selected because, the dependent variables are continuous

(Modulus of Elasticity E,) whilst the independent variables studied (wire dimension, prophylactic agent used and time of testing) were categorical.

The Generalized Linear Model was performed using SPSS software version 17. The analysis was performed separately for E, loading YS and unloading YS in each alloy type (NiTi and Cu NiTi). The Generalized linear model was used to study the effect of each of the studied independent variables (wire treatment, cross section and storage time) separately as well as their two-way interactions.

RESULTS

The results show a statistically significant decrease in the measured Modulus of Elasticity (E) the wires treated with both APF and MI Paste when compared with the control groups. In addition, the measured E values for the groups treated with MI Paste were significantly higher than those treated with APF. This change was less pronounced however still significant for the tested CuNiTi wires.

(Curves 1 to 6 and Table 3 to 7)

A representative sample of the generated load deflection curves





Delayed mododrtho st st 17x25 1 T.M

Table (3): GLM analysis showing significance of different variables on modulus of elasticity (E) of NiTi wires

	Source	Type III Sum of Squares	df	Mean Square	F	Sig.
	Corrected Model	43533.192ª	27	1612.340	24.132	.000
	Intercept	1573819.301	1	1573819.301	23555.686	.000
Main effect	Treat	948.850	3	316.283	4.734	.003
	Dim	31786.761	1	31786.761	475.759	.000
	Time	425.580	1	425.580	6.370	.012
	treat * dim	3508.815	3	1169.605	17.506	.000
Two way interaction	treat * time	655.968	2	327.984	4.909	.008
	dim * time	62.234	1	62.234	.931	.335

Significance level $P \le 0.05$

TABLE (4): Descriptive statistics showing effect of Time on Modulus of elasticity (E) of NiTi wires.

Time	Mean	Std. Error	95% Confidence Interval		
			Lower Bound	Upper Bound	
то	77.938	.653	76.652	79.224	
T1	74.057ª	.738	72.604	75.510	

	Source	Type III Sum of Squares	Df	Mean Square	F	Sig.
	Corrected Model	50833.005ª	14	3630.929	35.480	.000
	Intercept	814565.690	1	814565.690	7959.607	.000
	Treat	1500.952	3	500.317	4.889	.003
Main effect	Dim	39630.860	1	39630.860	387.257	.000
	Time	4308.285	1	4308.285	42.099	.000
Two way interaction	treat * dim	903.384	3	301.128	2.943	.035
	treat * time	1268.045	3	422.682	4.130	.008
	dim * time	1047.252	1	1047.252	10.233	.002
	Error	13815.553	135	102.337		
	Total	945686.670	150			
	Corrected Total	64648.558	149			

TABLE (5): GLM analysis showing significance of different variables on modulus of elasticity (E) of Cu NiTi wires

Significance level $P \le 0.05$

TABLE (6): Descriptive statistics showing effect of Time on Modulus of elasticity of Cu NiTi wires

Time	Mean	Std. Error	95% Confidence Interval		
			Lower Bound	Upper Bound	
ТО	80.830	1.131	78.593	83.067	
T1	71.850ª	1.209	69.459	74.241	

TABLE (7) Multiple comparison for wire dimensions

Pairwise Comparisons

Dependent Variable:E

		Mean Difference			95% Confidence Interval for Difference ^b	
(I) dim	(J) dim	(I-J)	Std. Error	Sig. ^b	Lower Bound	Upper Bound
1.00	2.00	32.709 ^{*,a}	1.656	.000	29.435	35.984
2.00	1.00	-32.709-*,c	1.656	.000	-35.984-	-29.435-

DISCUSSION

Compromised oral hygiene is a frequent complication with orthodontic fixed appliance therapy and can lead to enamel demineralization with the formation of white spot lesions and/ or decay, despite the presence of fluorides in the environment in toothpastes and mouth rinses. To address this potential treatment complication, orthodontists commonly prescribe a daily topical fluoride rinse and at the same time encourage in- office professional application of fluoride prophylactic agents.^{6,7}

The effects of such fluoridated prophylactic agents have been investigated, especially on titanium based alloys mainly from the view point of surface changes and discoloration⁵. There is limited information regarding the effect of prophylactic fluoride agents on the mechanical properties of wire alloys used today. For this reason, this in vitro study was conducted to evaluate the effect of two widely used prophylactic agents on mechanical properties of, nickel titanium, and copper nickel-titanium wires.

The APF (Acidulated phosphate fluoride) was chosen since it represents one of the most commonly used high concentration professionally applied fluorides^{17,18}. While MI Paste, which contains casein phosphoprotien amorphous calcium phosphate (CPP ACP) was used to represent a valid alternative to the conventional used fluorides.

This study also included a round cross-sectional wire group as well as a rectangular one. The reason that this selection was made can be attributed to the following; there is a difference in the manufacturing process of both round and rectangular wires¹⁹. These differences could have a significant effect on all the physical and mechanical properties of these wires. Furthermore different cross-sections have different surface area to volume ratios^{20,21,22}. This dissimilarity could have an effect on the way that the wire interacts with external environmental

influences. These influences include the effect of the prophylactic agents studied.

Topical application of fluoride especially in acidulated form has been reported to cause corrosion and deterioration of several properties of titanium based superelastic wires^{5,6,10}. These changes can be explained by the phenomena of hydrogen absorption and embrittlement that was previously documented for titanium based wires¹⁷.Hydrogen absorption and embrittlement of titanium-based alloys after fluoride exposure could be clarified by the fact that, although the surface oxide of titanium is known to be highly effective in reducing hydrogen penetration, ionizable fluoride compounds, such as sodium fluoride and hydrogen fluoride, activate the surface and can cause rapid corrosion. When titanium-based orthodontic wires are exposed to acidulated topical fluoride agents, it is suggested that hydrofluoric acid (HF) is produced according to the following equation (1) and dissolves the protective oxide layer on the surface of titanium and its alloys according to equations 2-45.

H3PO4 + 3NaF \rightarrow Na3PO4 + 3HF (1) Ti2O3 + 6HF \rightarrow 2TiF3 + 3H2O (2) TiO2 + 4HF \rightarrow TiF4 + 2H2O (3) TiO2 + 2HF \rightarrow TiOF2 + H2O (4)

It was stated previously that the copper component of the Cu NiTi wires appear to inhibit fluoride related degradation of the mechanical properties of those wires. However, he also stated that the copper component did not appear to be as effective in inhibiting surface pitting and corrosion. This finding is partial supported in this study with our finding that the E values for Cu NiTi wires showing a less substantial change.

Regarding the results for the NiTi testing, GLM main analysis interaction showed a significant effect for all tested independent variables on the measured E values. Multiple Comparisons Tests showed the two prophylactics caused a statistically significant reduction in the measured E values. Whist the comparison between the prophylactic agents showed no statistically significant difference between them.

Storage time had a statistically significant effect on the measured E values. The values of E measured at T1 were significantly less than those measured in T0.

As regards to Cu NiTi testing GLM analysis demonstrated significant effect for all the tested independent variables. The Multiple Comparison Tests exhibited a statistically significant reduction of the measured E values for both prophylactic agents compared to the control. The APF prophylactic agent showed statistically lower E values when compared with MI Paste.

In terms of time the E values measured at T1 were significantly lower than those measured at T0.

CONCLUSIONS

It could be concluded that NiTi wires are highly susceptible to dental prophylactic agents especially acidulated fluorides. The reduction in the values of the modulus of elasticity indicates the deterioration of the mechanical properties of the NiTi wires. Whereas, Cu NiTi wires were more resistant to the influence of the tested prophylactic agents

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