The effect of fluoride exposure on the load-deflection properties of superelastic nickel-titanium-based orthodontic archwires

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Background: It has been demonstrated that fluoride prophylactic agents may cause hydrogen absorption in NiTi wires and degrade their mechanical properties.
Aims: To investigate the effect of a fluoride mouthwash on load-deflection characteristics of three types of nickel-titanium-based orthodontic archwires.
Methods: Twenty maxillary 0.016 inch round specimens from each of the single-strand NiTi (Rematitan 'Lite'), multi-strand NiTi (SPEED Supercable) and Copper NiTi (Damon Copper NiTi) wires were selected. The specimens were kept in either 0.2% NaF or artificial saliva solutions at 37°C for 24 hours (N = 10). The wire load-deflection properties were measured by a Zwick testing machine, using a three-point bending test. An un-paired student's *t*-test, a one-way ANOVA and a Tukey post-hoc test were used to assess statistical significance.
Results: Immersion in NaF solution affected the load-deflection properties of NiTi wires. The unloading forces at 0.5 and 1.0 mm deflections were significantly lower in fluoride-treated specimens compared with the control groups ($p < 0.05$). Unloading forces at 1.5, 2.0 and 2.5 mm deflections were not statistically different between fluoride- and saliva-treated specimens ($p > 0.05$).
Conclusions: The results suggested that subjecting NiTi wires to fluoride agents decreased associated unloading forces, especially at lower deflections, and may result in delayed tooth alignment.
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Introduction

Wires made of nickel-titanium alloys are commonly used in orthodontic treatment as they possess valuable mechanical properties of superelasticity and shape memory. Their unique properties are attributed to a phase transformation between the austenitic form and martensitic structure of nickel-titanium, when wire is subjected to stress or temperature changes.[1,2] Superelasticity is characterised by the presence of a horizontal region or plateau range in the load-deflection graph, implying that a constant force value is exerted on teeth over long activation distances.[2,3] In recent years, ample attention has been paid to identifying superelastic archwires which produce a very light force in the plateau range. Light continuous forces are preferred in orthodontic treatment because they permit efficient tooth movement and cause less damage to the teeth or periodontium.[4,5] Multi-strand NiTi wires have been developed and proved to exert significantly lower force compared with conventional NiTi archwires.[6,7] In an attempt to deliver optimal forces to produce efficient tooth movement, Damon Copper NiTi (Cu NiTi) archwires were introduced. However, the possible degradation of the mechanical properties of multi-strand NiTi and Damon Cu NiTi wires remains a concern, as inadequate forces may be produced to achieve tooth alignment.

Maintaining good oral hygiene is a challenge in patients undergoing fixed orthodontic treatment. Appliances complicate conventional hygiene measures, resulting in significant plaque accumulation around bracket bases which may cause enamel demineralisation or white spot lesions. The prevalence of white spot lesions in patients undergoing fixed orthodontic treatment and who use fluoride toothpaste has been reported to be 13 to 75 per cent.[8,9] Because of its cariostatic effects, orthodontists often recommend that patients use complementary fluoride agents, in addition to fluoride toothpaste, to reduce the incidence of decalcification.

A protective layer of titanium oxide on the surface of titanium alloys is easily degraded in fluoride solutions, thereby exposing the underlying alloy to the oral environment. Upon exposure, hydrogen ions from the ambient air or aqueous-based solutions, propagate through the alloy via interstitial locations, dislocations and grain boundaries to react with lattice atoms.[10-12] Hydrogen absorption, as a result of fluoride-related disruption of the titanium oxide layer, has been considered to produce degradation of the mechanical properties[12-15] and reduce corrosion resistance[12,16,17] of NiTi wires. Furthermore, hydrogen absorption may cause discolouration and an alteration in the surface structure of titanium-based alloys.[18]

The effects of fluoride prophylactic agents on the superelastic properties of NiTi archwires have not been evaluated. The aim of the present study was to determine the load-deflection properties of single-strand NiTi, multi-strand NiTi and Damon Cu NiTi wires after immersion in a 0.2% neutral fluoride mouthwash, compared with a pH 7 artificial saliva solution.

Materials and methods

The test sample comprised three types of nickel-titanium-based orthodontic wires: Rematitan 'Lite' (Dentaurum, Ispringen, Germany), SPEED Supercable (Strite Industries, Cambridge, ON, Canada) and Damon Cu NiTi (Ormco, Glendora, CA, USA). These wires are available as a single-strand or multi-stranded, as listed in Table I. Preformed maxillary 0.016 inch round wires were selected for this study.

Twenty specimens of each wire type were cut in lengths of 30 mm from the straight, posterior parts of manufacturer's preformed archwires. The specimens were immersed in either 0.2% neutral NaF solution or artificial saliva of pH 7 and kept at 37°C for 24 hours. The artificial saliva consisted of 1 g of sodium carboxymethylcellulose, 4.3 g of xylitol, 0.1 g of potassium chloride, 5 mg of calcium chloride, 40 mg of potassium phosphate, 1 mg of potassium thiocyanate and 100 g of distilled deionized water.[19] Immediately prior to mechanical testing, the wires were removed from the solutions, rinsed with water and air dried. Subsequently, the specimens were inserted into a plastic phantom head jaw, on which two 0.018 inch standard edgewise molar tubes (Dentaurum, Ispringen, Germany) were bonded on teeth 11 and 13. Accurate tube alignment was achieved by inserting a full-size archwire into the rectangular tubes before resin curing. A 15.5 mm distance was selected between the midpoint of the tubes to represent the average tooth dimensions for a maxillary permanent male dentition.

A Zwick Testing Machine (Zwick GmbH and Co, Ulm, Germany) fitted with a 250 kg load cell was used to apply a modified three-point bending test to assess the load-deflection properties of the wires. The mid-point of the wire specimen was deflected using the movable part of the testing machine which had a groove to accommodate the wire. A crosshead speed of 1 mm/min was chosen to deflect the wire a maximum distance of 3 millimetres. Figure 1 shows a wire specimen in its partially restrained and deflected state within the system. All tests were performed at a constant temperature chamber of 37°C. The water temperature was controlled by a thermometer to an

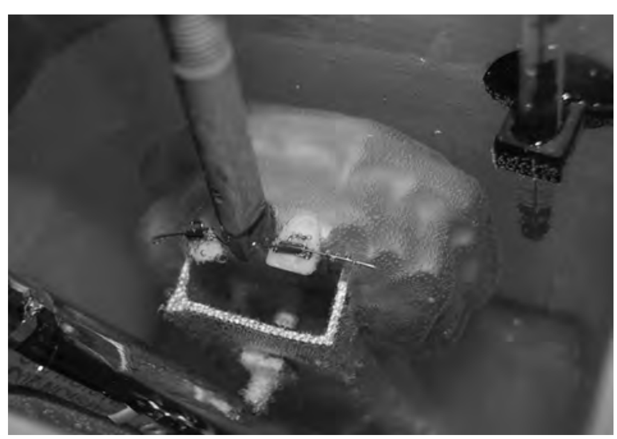

Figure 1. Close up view of the modified three-point bending model on the phantom head jaw with the specimen deflected.

accuracy of ± 0.5°C. The loading and unloading forces were registered by the load cell and transmitted to a computer using an analogue/digital convertor. Load-deflection curves were produced and drawn for all specimens. Unloading forces were used for statistical analyses, since unloading of the wire provides the forces necessary for tooth movement.

Forces generated at intervals of 0.5 mm deflection during unloading (from 2.5 mm to 0.5 mm) were determined to represent the load-deflection properties of each specimen. After the normal distribution of the data was confirmed by the Kolmogorov-Smirnov test and the equality of variances by the Levene's test, a one-way ANOVA was used to analyse load differences between the three wires. A Tukey post hoc pair-wise comparison test was performed to identify significance in between-group differences. The statistical difference in unloading forces of each wire between NaF and the control artificial saliva solutions was detected by an unpaired *t*-test. A *p* value less than 0.05 ($p \leq 0.05$) was considered to be statistically significant.

Results

Table II presents means and standard deviations of forces at intervals of 0.5 mm deflection during unloading for the three wires in NaF and artificial saliva solutions. In general, the maximum force in either NaF or artificial saliva solutions was exerted by Rematitan 'Lite' supplied by Dentaurum and the minimum force was exerted by Supercable supplied by Strite Industries. The load-deflection graphs of Rematitan 'Lite', Supercable and Damon Cu NiTi wires in NaF and artificial saliva solutions are shown in Figures 2 to 4, respectively. The unloading sections of the load-deflection graphs of all wires in both solutions contained a nearly horizontal region or plateau range, where deactivation occurred at nearly constant force values. The load-deflection graphs of NiTi wires exposed to NaF solution had notable differences compared with those immersed in artificial saliva solution (Figures 2 to 4).

Unpaired *t*-tests substantiated several significant differences in load-deflection characteristics of the three wires between NaF and artificial saliva solutions ($p < 0.05$). NaF-treated specimens exhibited statistically lower forces compared with saliva-treated specimens at 1.0 and 0.5 mm deflections during unloading (Table II). For example, unloading forces

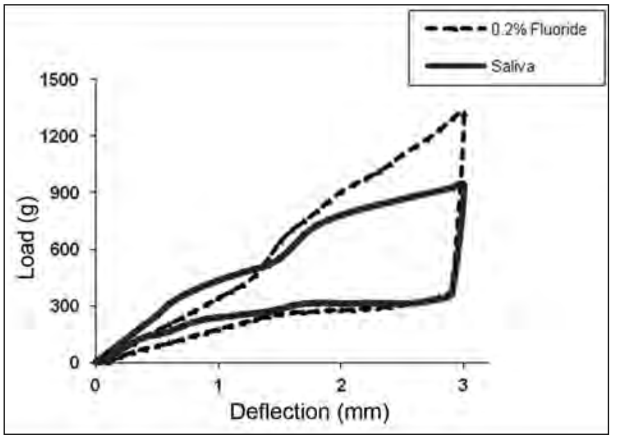

Figure 2. Comparison of load-deflection curves of the single-strand NiTi wire (Rematitan 'Lite') after immersion in artificial saliva and 0.2% NaF solutions.

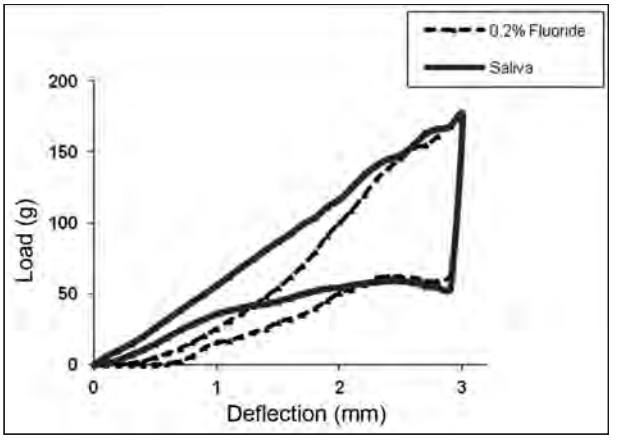

Figure 3. Comparison of load-deflection curves of the multi-strand NiTi wire (Supercable) after immersion in artificial saliva and 0.2% NaF solutions.

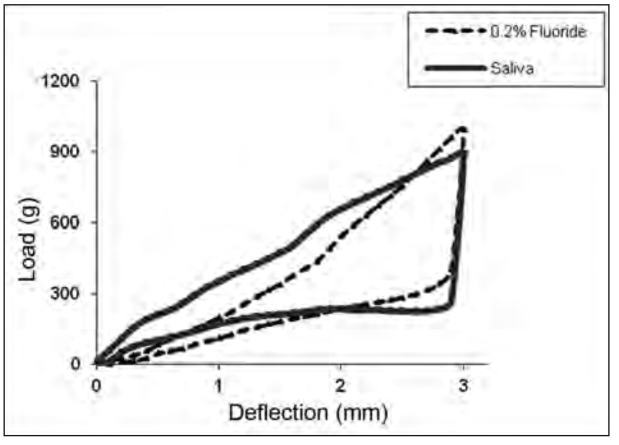

Figure 4. Comparison of load-deflection curves of the Cu NiTi wire (Damon Cu NiTi) after immersion in artificial saliva and 0.2% NaF solutions.

Table I. List of different nickel-titanium archwires tested in this study (0.016 inch round, maxillary arch only).

Commercial name	Composition	Number of strands	Manufacturer
Rematitan 'Lite'	NiTi	Single-strand	Dentaurum
Supercable	NiTi	Multi-strand	Speed
Damon Copper NiTi	Cu NiTi	Single-strand	Ormco

Table II. Means and standard deviations (g) and the results of unpaired t-tests of loads at intervals of 0.5 mm deflection during unloading for Rematitan 'Lite', Supercable and Damon Copper NiTi wires in NaF and artificial saliva solutions.

Deflection (mm)	Rematitan 'Lite'			Supercable			Damon Copper Niti		
	Saliva	NaF		Saliva	NaF		Saliva	NaF	
	Mean ± SD	Mean ± SD	*p*	Mean ± SD	Mean ± SD	*p*	Mean ± SD	Mean ± SD	*p*
2.5	314 ± 15.3	313 ± 45.3	0.950	59 ± 7.1	61 ± 12.2	0.756	224 ± 12.7	281 ± 19.9	0.059
2.0	315 ± 19.3	281 ± 42.3	0.239	54 ± 6.1	51 ± 12.2	0.731	234 ± 9.1	236 ± 20.9	0.892
1.5	286 ± 14.2	260 ± 21.3	0.077	44 ± 5.1	30 ± 11.2	0.087	210 ± 7.1	179 ± 32.6	0.167
1.0	241 ± 19.3	179 ± 29.0	0.005	36 ± 5.6	16 ± 7.6	0.007	169 ± 4.0	108 ± 29.0	0.005
0.5	151 ± 12.2	92 ± 25.5	0.002	15 ± 3.5	0 ± 2.0	<0.0001	100 ± 3.5	43 ± 19.4	<0.0001

Table III. Results of ANOVA for comparison of force levels of Rematitan 'Lite', Supercable and Damon Copper NiTi wires at different deflections after immersion in artificial saliva and 0.2% NaF solutions.

	2.5	2.0	1.5	1.0	0.5
	p	*p*	*p*	*p*	*p*
Saliva	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001
NaF	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001

at a deflection of 1.0 mm were 241 g (artificial saliva) versus 179 g (NaF) for Rematitan 'Lite', 36 g (artificial saliva) versus 16 g (NaF) for Supercable, and 169 g (artificial saliva) versus 108 g (NaF) for Damon Cu NiTi specimens. This indicates that immersion of Rematitan 'Lite', Supercable, and Damon Cu NiTi wires in NaF solution caused a 26, 55, and 36% reduction of forces, respectively, compared with saliva-treated specimens.

The results of ANOVA (Table III) demonstrated significant differences in force levels of the three wires in either NaF or artificial saliva solutions ($p < 0.0001$). The Tukey post-hoc test (Tables IV and V) showed that in both media, Rematitan 'Lite' possessed significantly greater force than Damon Cu NiTi, and Damon Cu NiTi retained significantly greater force than Supercable at all deflections ($p < 0.05$).

Discussion

Titanium-based alloys spontaneously form a nanometer thickness layer of titanium oxide, one millisecond after exposure to air, in a process called passivation.[18,20] However, this protective layer may be degraded following exposure to fluoride prophylactic agents. A small amount of fluoride in an acidic environment can form hydrofluoric acid (HF), which easily dissolves the protective oxide layer on the surface of titanium-based alloys to form titanium fluoride or titanium oxyfluoride,[10,12,18,21,22] which in turn, causes hydrogen absorption within the alloy. The resulting fracture or degradation of mechanical properties of titanium-based alloys caused by hydrogen ions is generally termed hydrogen embrittlement.[13-14,23] Although hydrogen embrittlement takes place more easily in acidic rather than neutral environments, Yokoyama

Table IV. Ranking of forces (g) generated by Rematitan 'Lite', Supercable and Damon Copper NiTi wires at different deflections after immersion in artificial saliva solution.

	Rematitan 'Lite'		Supercable		Damon Copper NiTi	
Deflection	Mean	Rank	Mean	Rank	Mean	Rank
2.5	314	3	59	1	224	2
2	315	3	54	1	234	2
1.5	286	3	44	1	210	2
1	241	3	36	1	169	2
0.5	151	3	15	1	100	2

Table V. Ranking of forces (g) generated by Rematitan 'Lite', Supercable and Damon Copper NiTi wires at different deflections after immersion in 0.2% NaF solution.

	Rematitan 'Lite'		Supercable		Damon Copper NiTi	
Deflection	Mean	Rank	Mean	Rank	Mean	Rank
2.5		3	61	1	281	2
2.0	281	3	51	1	236	2
1.5	260	3	30	1	179	2
1.0	179	3	16	1	108	2
0.5	92	3	0	1	43	2

et al.[15] indicated that hydrogen absorption of titanium-based wires can also take place in neutral fluoride agents. Kaneko et al.[23] observed corrosive pits on the surface of titanium-based specimens in both neutral NaF and acidulated phosphate fluoride (APF) solutions. Walker et al.[12] reported that neutral and acidulated fluoride agents produced a significant reduction of the NiTi wire unloading properties, although the effect was more pronounced with the acidulated fluoride agent.

In the present study, a modified three-point bending test was used to assess the load-deflection properties of various archwires after exposure to a fluoride prophylactic mouthwash. This test provided reproducibility and simplified comparison with other studies,[24-26] but its appropriateness as the standard wire test has been questioned by previous authors.[27,28] To eliminate binding and friction that affects the force delivery properties of orthodontic wires, molar tubes were bonded on central incisor and canine teeth instead of brackets.[29-32] As in most previous studies[30,33-35] the tests were performed at 37°C water temperature to closely approximate oral conditions.

The force level of the tested multi-strand NiTi wire was very low, ranging from 59 g at 2.5 mm deflection to 15 g at 0.5 mm deflection for specimens immersed in the artificial saliva solution. The force range of this wire after storage in the 0.2% NaF solution was between 61 g and 0 g at 2.5 and 0.5 mm deflections, respectively. The force delivered by the multistrand wire was significantly lower than those exerted by Damon Cu NiTi and conventional NiTi wires, which corroborated the findings of previous studies.[7,36] The optimal force range for orthodontic tooth movement has been considered to be 30-80 g, which stimulates cellular activity without overtly exceeding capillary blood pressure.[37] Therefore, the force magnitude of fluoride-treated Supercable specimens at lower deflections appeared to be lower than accepted optimal force levels for tooth movement, which may result in treatment delay.

The force level of saliva-treated Damon Cu NiTi specimens varied from 224 g at 2.5 mm deflection to 100 g at 0.5 mm deflection, while NaF-treated specimens demonstrated a force range between 281 g and 43 g at 2.5 and 0.5 mm deflections, respectively. Damon Cu NiTi exerted significantly lower forces than conventional NiTi wire, which agreed with a previous study.[7] The statistically significant force reduction of this wire at 1.0 and 0.5 mm unloading deflections suggested that a decrease in its clinical performance could be expected upon fluoride exposure.

The force level of single-strand NiTi wire was several times greater than other types of wire and ranged from 314 to 151 g in saliva-treated specimens, and 313 to 92 g in NaF-treated specimens, at 2.5 and 0.5 mm deflections, respectively. The degradation in force magnitude of this wire after exposure to fluoride agents may cause little clinical concern because the degraded wire would still exert forces that are greater than the optimal force range for tooth alignment. It should be noted that the forces measured in a three-point bending test are not directly transferable to the clinic. Therefore, greater emphasis should be placed on the rank order of the wires than on the force values expressed in the data tables.[28]

After exposure to the 0.2% neutral fluoride mouthwash, Supercable and Damon Cu NiTi wires experienced a clinically remarkable decrease in unloading forces at lower deflections which may result in delayed tooth alignment. To counteract the degradation effects, it is recommended that clinicians religate NiTi wires frequently in the alignment stage of orthodontic treatment for patients undergoing strict fluoride regimens. In this way, the wire stiffness in the load-deflection graph will increase and consequently a greater force level will be exerted on the teeth at lower deflections.[1,3,4] It has also been recommended that careful application of fluoride containing products be observed in orthodontic patients to minimise unwanted damage on orthodontic wires.[10,13,16,17,38] Schiff and coworkers[16] suggested that patients rinse their mouth after the use of fluoride mouthwashes to limit any remaining topical action of the fluoride which could corrode titanium alloys; however, this may decrease the dental benefits of fluoride agents.

Walker et al.[12] reported that the application of acidic and neutral fluoride treatments had no significant effect on Cu NiTi elastic modulus and yield strength (YS), but significantly decreased the unloading modulus and unloading YS of NiTi orthodontic wire, compared with distilled-water control treatment. It was assumed that the copper component in the Cu NiTi archwires partially inhibited the activity of hydrofluoric acid and therefore prevented fluoride-related degradation in the mechanical properties of Cu NiTi wires.[12] Ramalingam and coworkers[39] reported that the unloading modulus of elasticity of NiTi archwires retrieved from patients who used a fluoride gel for 30 days decreased significantly, while the mechanical properties of Cu NiTi archwires were not affected by fluoride agents. Schiff et al.[16,17] indicated that NiTi wires were more susceptible than Cu NiTi wires to corrosion. The findings of the present study are in contrast with those of Walker et al.,[12] Ramalingam et al.[39] and Schiff et al.,[16,17] because Damon Cu NiTi wire was shown to experience a significant decrease in unloading force at lower deflections.

In the present study, the 0.2% neutral NaF solution had a significant effect on load-deflection characteristics of NiTi archwires. The pH of neutral fluoride agents is approximately 7. The aggressive potential of fluoride prophylactic agents on titanium-based alloys increases with the rise of fluoride concentration and fall of pH value.[40-43] Therefore, a more profound decrease in unloading forces may be expected by the exposure of NiTi wires to acidic fluoride agents or higher fluoride concentrations.

The time of fluoride application in the present study was chosen according to previous reports involving 1 to 3 days of exposure.[10,18,21,42,44] Although patients use the 0.02% fluoride mouth wash one minute per week, the real time of fluoride exposure may be longer, since patients are instructed to apply fluoride before bedtime and to refrain from rinsing their mouths for at least 30 minutes thereafter. In addition, in vivo fluoride exposure may be greater due to the application and use of fluoride gels and fluoridated toothpaste or water.[12,22]

Care should be taken in interpreting the results of this in vitro study, as the oversimplified laboratory methods may not replicate the complex oral environment.[45] Further clinical research is recommended on the clinical efficiency of orthodontic archwires subjected to various fluoride agents.

Conclusions

1. Following fluoride exposure, there was a significant decrease in unloading forces of superelastic NiTi wires at lower deflections, which would likely result in delayed tooth alignment.
2. It is recommended that clinicians religate NiTi wires more frequently in the initial stage of orthodontic treatment in patients who regularly use fluoride prophylactic agents. This would ensure the delivery of sufficient force for tooth alignment.

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