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ABSTRACT

Background: Hydrogen absorption and related degradation in the mechanical properties of Ni-Ti based orthodontic wires has been demonstrated following exposure to fluoride prophylactic agents. This study was designed to investigate the effects of three fluoride containing agents on the load deflection characteristics of heat activated nickel titanium arch wires during unloading phase.

Material and method: Eighty specimens of heat activated nickel titanium arch wires were obtained from Ortho Technology Company, half of which had a 0.016 inch round and 0.019x0.025 rectangular. Ten specimens from both wire size were immersed in one of the tested fluoride prophylactic agents (neutral sodium fluoride gel, stannous fluoride gel or phos-flur mouth rinse) or in the controlled medium “normal saline”, and incubated at 37°C for sixty minutes.

A Wp 300 universal material testing machine was modified and used to perform a three point bending test in a water path at 37°C ±1°C. The statistical difference between the different agents were analyzed using ANOVA and LSD tests.

Results: The unloading forces at 0.5, 1.0, and 1.5 mm where significantly reduced especially in neutral sodium fluoride treated specimens.

Conclusion: Based on the results founded in this study it might be preferred to use prophylactic agent with the least fluoride ions concentration. It can be concluded that the tested agents have only a limited effect on the load deflection behavior of the heat activated Ni-Ti wires, in a way that they do not have a clinically significant effect on the mechanical behavior of these wires.

Key words: Fluorides, load-deflection, Heat activated Ni-Ti wires.

INTRODUCTION

Orthodontic wires made from titanium alloys provide light continuous force with large amount of activation for long periods. This makes them extremely useful as initial or intermediate wires between the first alignment and finishing stages of treatment(1).

Heat-activated nickel titanium wires have been gaining popularity in the orthodontic practice during the last decade. These so called third generation wires have been marketed with clinically useful shape memory property which is the capability of Ni-Ti wires to be plastically deformed in their martensitic phase, in addition to the low stiffness, high spring back and super elasticity (1,2).

Fluoride prophylactic agent, such as acidulated phosphate fluoride (APF), have been used extensively to prevent demineralization or re-mineralization of white spot lesions around orthodontic brackets and bands; however, the fluoride ions in the prophylactic agents have been reported to cause corrosion and discoloration of titanium and its alloys (3). Degradation in the mechanical properties lead to a reduction in appropriate orthodontic force, thereby causing delayed straightening of irregular teeth (4).

This is an in vitro study is to evaluate the effect of three different fluoride agents on the load deflection of heat-activated nickel titanium arch wires.

MATERIALS AND METHODS

The samples comprised eighty pieces of 4cm length obtained by cutting the straight, posterior portion of preformed upper heat-activated nickel titanium arch wire using a cutter. Forty pieces were 0.016” and forty pieces were 0.019x0.025” (Ortho Technology Co., CA, U.S.A.). These samples were divided into four groups, each group contains 20 pieces (10 pieces of 0.016” and 10 pieces 0.019x0.025”) according to immersion medium:
1) Control medium (normal saline 0.9% w/v pH=7).
2) 0.4% Stannous fluoride gel (Dental Technologies alpha-dent, Lincolnwood, Illinois, U.S.A with pH=3.3)
3) 0.044% w/v Phos-flur mouth rinsed (Colgate Oral Pharmaceutical, New York, U.S.A with pH=4.2)
4) 1.1% Neutral sodium fluoride gel (DentMat Holdings, Lompoc, California, U.S.A with pH=7).

All samples were incubated at 37°C in inert plastic tubes of 10ml capacity for sixty minutes (60 minutes=1 minutes per day topical fluoride
application for two months). Then the samples were removed from their respective test media washed with normal saline and placed in a new, clean, and individually labeled plastic tubes before mechanical testing.

The three point bending test was carried out to test the load deflection characteristics of the selected arch wires. The samples were mounted into a three point bending test fixture (stainless steel jig with two barreled rods set 15 mm apart) the mid portion of the wire were loaded to 2mm deflection by rotating the hand wheel of the WP300 universal testing machine (G.U.N.T. Gerätebau GmbH, Hamburg, Germany) in clock wise direction then very gently unwind the hand wheel in counter clock direction to unload to zero deflection. For statistical analysis the unloaded forces at 1.5, 1.0, 0.5 mm were used since unloading phase of the wire represent the necessary forces to achieve tooth movement. To simulate aqueous oral environment the test was carried out in a water bath at 37°C ±1°C the temperature was controlled by using a digital thermometer.

One way analysis of variance (ANOVA) was used to examine whether any significant difference at $p<0.05$ exist between the four tested groups. Further, LSD was used to compare among tested groups.

**RESULTS**

Table 1 showed the means and standard deviations of forces at intervals of 0.5 mm deflection during unloading for 0.016" and 0.019x0.025" heat activated nickel titanium arch wire. F-test by ANOVA table showed that there was statistically a highly significant difference in the load deflection of 0.016" and 0.019x0.025" heat activated nickel titanium arch wires immersed in different fluoride agents during unloading at 1.5, 1.0, 0.5 mm $p<0.001$.

Table 2 showed the results of LSD after ANOVA for 0.016" and 0.019x0.025" heat activated arch wire. The load deflection graphs for heat-activated nickel titanium arch wires after being immersed in tested agents in comparison with the control group are presented in figure (1).

**Table 1: Descriptive statistics of load in (gm.) during unloading phase and groups' difference for HANT arch wires.**

<table>
<thead>
<tr>
<th>Arch wires dimension</th>
<th>Deflection (mm.)</th>
<th>NaCl</th>
<th>SnF2</th>
<th>APF</th>
<th>NF</th>
<th>Media difference</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Mean</td>
<td>SD</td>
<td>Mean</td>
<td>SD</td>
<td>Mean</td>
</tr>
<tr>
<td>0.016&quot;</td>
<td>1.5</td>
<td>64.25</td>
<td>1.42</td>
<td>61.9</td>
<td>1.20</td>
<td>60.15</td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>47.5</td>
<td>1.18</td>
<td>43.4</td>
<td>2.84</td>
<td>43.5</td>
</tr>
<tr>
<td></td>
<td>0.5</td>
<td>40.1</td>
<td>2.16</td>
<td>38.35</td>
<td>2.22</td>
<td>38.6</td>
</tr>
<tr>
<td>0.019x0.025&quot;</td>
<td>1.5</td>
<td>174</td>
<td>3.64</td>
<td>173.20</td>
<td>6.72</td>
<td>171.60</td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>113.20</td>
<td>4.26</td>
<td>113.60</td>
<td>6.88</td>
<td>111.10</td>
</tr>
<tr>
<td></td>
<td>0.5</td>
<td>106.65</td>
<td>6.05</td>
<td>108.95</td>
<td>5.44</td>
<td>107.70</td>
</tr>
</tbody>
</table>

**Table 2: LSD after ANOVA for 0.016" and 0.019x0.025" heat activated arch wire**

<table>
<thead>
<tr>
<th>Deflection (mm.)</th>
<th>Groups</th>
<th>Mean Difference</th>
<th>$p$-value</th>
<th>Mean Difference</th>
<th>$p$-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.016&quot;</td>
<td>NaCl</td>
<td>SnF2</td>
<td>2.35</td>
<td>0.055 (NS)</td>
<td>0.80</td>
</tr>
<tr>
<td></td>
<td></td>
<td>APF</td>
<td>4.1</td>
<td>0.001 (HS)</td>
<td>2.40</td>
</tr>
<tr>
<td></td>
<td></td>
<td>NF</td>
<td>6.15</td>
<td>0.000 (HS)</td>
<td>15.90</td>
</tr>
<tr>
<td></td>
<td>SnF2</td>
<td>APF</td>
<td>1.75</td>
<td>0.148 (NS)</td>
<td>1.60</td>
</tr>
<tr>
<td></td>
<td></td>
<td>NF</td>
<td>3.8</td>
<td>0.003 (HS)</td>
<td>15.10</td>
</tr>
<tr>
<td></td>
<td>APF</td>
<td>NF</td>
<td>2.05</td>
<td>0.092 (NS)</td>
<td>13.50</td>
</tr>
<tr>
<td></td>
<td></td>
<td>SnF2</td>
<td>4.1</td>
<td>0.001 (HS)</td>
<td>-0.40</td>
</tr>
<tr>
<td></td>
<td>NaCl</td>
<td>APF</td>
<td>4</td>
<td>0.001 (HS)</td>
<td>2.10</td>
</tr>
<tr>
<td></td>
<td></td>
<td>NF</td>
<td>6</td>
<td>0.000 (HS)</td>
<td>16.00</td>
</tr>
<tr>
<td></td>
<td>SnF2</td>
<td>APF</td>
<td>-0.1</td>
<td>0.929 (NS)</td>
<td>2.50</td>
</tr>
<tr>
<td></td>
<td></td>
<td>NF</td>
<td>1.9</td>
<td>0.096 (NS)</td>
<td>16.40</td>
</tr>
<tr>
<td></td>
<td>APF</td>
<td>NF</td>
<td>2</td>
<td>0.080 (NS)</td>
<td>13.90</td>
</tr>
<tr>
<td></td>
<td></td>
<td>SnF2</td>
<td>1.75</td>
<td>0.112 (NS)</td>
<td>-2.30</td>
</tr>
<tr>
<td></td>
<td>NaCl</td>
<td>APF</td>
<td>1.5</td>
<td>0.171 (NS)</td>
<td>-1.05</td>
</tr>
<tr>
<td></td>
<td></td>
<td>NF</td>
<td>3.75</td>
<td>0.001 (HS)</td>
<td>15.10</td>
</tr>
<tr>
<td></td>
<td>SnF2</td>
<td>APF</td>
<td>-0.25</td>
<td>0.817 (NS)</td>
<td>1.25</td>
</tr>
<tr>
<td></td>
<td></td>
<td>NF</td>
<td>2</td>
<td>0.071 (NS)</td>
<td>17.40</td>
</tr>
<tr>
<td></td>
<td>APF</td>
<td>NF</td>
<td>2.25</td>
<td>0.043 (HS)</td>
<td>16.15</td>
</tr>
</tbody>
</table>

NS: $p>0.05$ (Not significant) S: $p<0.05$ (Significant) HS: $p<0.001$ (Highly significant).
DISCUSSION

Within one millisecond exposure to air, titanium-based alloys form a nanometer thickness layer of titanium oxide $10-20\text{nm}$, in a process called passivation \(^{(5,6)}\). However, this protective layer may be degraded following exposure to fluoride prophylactic agents. Topical fluoride agents have been reported to cause corrosion of titanium based arch wires \(^{(4,5,7-10)}\). It should be understood that the fluoride related effect depends on concentration of fluoride ions in the agent being used, the pH level of the agent, the duration of immersion, and the wires manufacturing characteristics \(^{(11)}\).

In the current study, the three point bending test is conducted. It is a standardized testing method useful for purely theoretical evaluations, offers a high level of reproducibility and allows comparison with other studies \(^{(12-14)}\). The beam tests was carried out using a jig machined from stainless steel with two barreled rods that set 15 mm apart to simulate a typical interbracket span \(^{(15)}\).

In the current study, a commercially available heat activated nickel-titanium archwires were tested with two cross sections 0.016" and 0.019x0.025". These arch wire gauges were selected because of their clinical popularity for aligning and leveling phase to generate low force levels due to material properties which was adopted by the MBT method \(^{(16)}\). Wire deflections of 2mm and then the unloading forces at 0.5mm interval were selected because of its possible occurrence under clinical conditions \(^{(2)}\).

In the current study, the fluoride agents that were used differ in their fluoride ion concentration and pH value and according to manufacturer instructions they used for one minute per day topical application. The NaCl were used as a control medium because Ni-Ti based arch wires have high corrosion resistance in NaCl solution \(^{(17)}\). NaCl has adopted as a control medium by previous studies \(^{(18,19)}\).

The results of the current study are in agreement with other findings \(^{(23-25)}\). Sabaneet \(^{(23)}\) and Koushiket al. \(^{(24)}\) found significant reduction in unloading mechanical properties of Ni-Ti and Cu Ni-Ti following exposure to fluoride agents after ninety minutes immersion time.

Ahariet et al. \(^{(25)}\) found significant reduction in the unloading forces at lower deflection following immersion of Ni-Ti and Cu Ni-Ti in 0.2% Sodium fluoride solution for 24 hour.

The results of the current study are in contrast to the finding by others \(^{(4,20-22)}\). Walker et al. \(^{(4)}\) reported that the application of acidic and neutral fluoride treatments have no significant effect on Cu Ni-Ti (copper Ni-Ti that show a thermal properties) mechanical properties compared with distilled-water control treatment, but a reduction in the unloading mechanical properties of Ni-Ti wires was observed. It was assumed that the copper component in the Cu Ni-Ti archwires partially inhibit the activity of hydrofluoric acid; therefore, prevent fluoride related degradation in the mechanical properties of Cu Ni-Ti wires. Walker and his coworkers have noticed surface corrosion in Ni-Ti and Cu Ni-Ti arch wires in their study.

Ramalingam et al. \(^{(22)}\) reported that the mechanical properties of Cu Ni-Ti archwires retrieved from patients who used a fluoride gel and phos-flur rinse for 30 days were not affected by fluoride agents but Ni-Ti wires had a reduction in the unloading force especially in gel group.

Schiff et al. \(^{(20,21)}\) indicated that Ni-Ti wires were more susceptible to corrosion than Cu Ni-Ti wires.

In the current study, it seems that the fluoride related hydrogen embrittlement of titanium based
alloys affecting the wire unloading –related phase shift\(^{(4,10,26)}\). Hydrogen absorption and subsequent diffusion through the interstitial sites, dislocations, and grain boundaries reacting with lattice atoms forming titanium hydride which form a body centered tetragonal structure could interfere with the lattice's ability to undergo the unloading phase shift from the martensitic form to the austenitic form. This might be considered to be the cause of related degradation of mechanical properties of the alloy \(^{(4,24,27)}\). This phenomenon might account for statistically significant differences in the unloading properties of the wires.

In rectangular wires the load deflection mean during unloading was significantly reduced in all selected deflection points in NF gel group which has the highest concentration of fluoride among the test groups. The same condition was noticed in the round arch wires, NF gel caused reduction in the unloading forces at all deflection points. It also noticed that the SnF\(_2\)gel and APF rinse influenced on the round wires more than the rectangular wires. This could be attributed to the fact that the absorbed hydrogen in titanium alloys diffuses from the surface inward even at room temperature, and diffusion distance depends on the coefficient of hydrogen diffusion in materials; therefore, for thinner nickel-titanium and beta titanium wires, degradation in performance caused by hydrogen absorption probably occurs for a short immersion time\(^{(26)}\), which is in consistent with the results of the current study.

In the current study, the reduction in the unloading forces might not be large enough to be clinically significant, since the wire was still exerting force levels within the optimal force range to produce tooth movement. The statistically significant difference occurred only after 60 minutes of fluoride exposure. In clinical situation, the real exposure time may be longer than 60 minutes because the patients are usually instructed to apply the fluoride agent before bed time and not to rinse their mouths, eat or drink for at least thirty minutes thereafter. Also, the orthodontic arch wire could be kept in mouth for longer duration which increase the overall exposure time.

In the current study, the margin of difference in the load values in fluoride agents was in-between 6gm to 16gm compared to control group. Although there was a statistically significant difference the amount of reduction, clinically, in the load was small. Therefore, using fluoride agents seems to be suitable when using heat activated nickel titanium wires especially agents with lower concentration of fluoride ion such as phos-flur rinse and stannous fluoride gel agents.

REFERENCES


**Summary:**

Aim: The aim of this study was to investigate the effect of three fluoride-containing products on the deflection of NiTi archwires during the debonding process.

**Materials and Methods:**

Eighty NiTi archwires were divided into four groups: Group 1 (control) was immersed in water, and Groups 2, 3, and 4 were immersed in fluoride-containing products (methylene blue fluoride paste, silver fluoride gel, or fluoride mouthwash), respectively. Each group consisted of 20 samples. The samples were subjected to a cyclic loading test to measure the deflection at 0 and 0.5 mm. Data were analyzed using ANOVA and LSD tests.

**Results:**

The results showed that the use of fluoride-containing products significantly reduced the deflection of NiTi archwires compared to the control group.

**Conclusion:**

The use of a low concentration of fluoride-containing products may be beneficial in reducing the deflection of NiTi archwires during debonding.