EFFECTS OF THERMOCYCLING AND PH ON ULTRASTRUCTURE CHARACTERISTICS OF NITI ARCH WIRES

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Abstract

Objectives: Evaluating the effects of saliva pH and thermocycling on the surface structural and mechanical characteristics of NiTi arch wires.

Materials and methods: The method of this study was *in vitro* or experimental. Forty samples of NiTi wires of 5 cm length and 0.016 inch diameter were procured from “Ormco Company” and used in this study. The samples were randomly divided into four groups with different conditions including pH of 4.5 and 7.4 with and without thermocycling. Thermocycling was performed in 500 cycles from 5 to 55 ◦C in water. The surface structure and mechanical properties were investigated by a profilometer (RTD-200 Portable Surface Rough-ness tester, New Star, Australia) and Instron machine, respectively. The obtained data were analyzed using ANOVA following by Post Hoc test.

Results: The highest surface-roughness was achieved in pH 4.5 with thermocycling (0.50±0.09 μm/Ra); while the lowest roughness was estimated to be 0.20±0.04 μm/Ra in the group of pH 7.4 without thermocycling (P<0.01). Moreover, the highest and lowest stiffness value was observed in pH 7.4 under non-thermocycling (15.2±0.6 g/mm2) and in pH 4.5 with thermocycling (8.3±0.3 g/mm2, P<0.001), respectively.

Conclusion: Both acidic pH and thermocycling process considerably influence and negatively impact the properties of NiTi alloy arch wires. Based on our results, the lowest changes in the surface structure and mechanical characteristics of NiTi wires were observed in pH 7.4 under non-thermocycling condition.

Keywords: orthodontics; surface roughness; stiffness; saliva pH; thermocycling; NiTi arch wires
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Introduction

One of the most crucial factors in orthodontic treatments is to control the forces related to orthodontic appliances [1]. This factor is often affected by the frictional forces between two important components: arch wire and bracket [2,3]. Surface properties such as roughness, hardness, and topography of orthodontic arch wires can be affected by frictional forces. Also, the aesthetics of dental products, biocompatibility and corrosion potential are often determined by their surface properties [4]. For this purpose, different alloy based wires are available with required features [5]. However, during different steps of treatments, these wires go through different stresses, and none of them is fully optimal for orthodontic applications [6]. In this context, nickel-titanium (NiTi) is one of the suitable alloys, which was introduced for the first time into clinical uses in 1972 [7]. Over the past decades, the use of NiTi alloy has increased in dental-related industries, especially to develop arch wires [1]. However, many studies have reported Ni release from NiTi appliances under various static and dynamic conditions, which essentially lead to potential toxic effects in the system [8,9].

During active orthodontic treatments, these arch wires are employed only for a relatively short duration with an average span of around 2 years [10]. In the oral environment, there are several factors including thermal, pH, enzymatic activities and microbial flora that can affect the performance of these arch wires [11,12]. In addition, one of the important aspects of the investigation on this arch wire is corrosion. The corrosion processes are typically influenced by several conditions of oral activities including (i) the temperature, (ii) saliva factors such as the quantity and pH, (iii) some enzymatic actions, and (iv) the chemical and physical properties of foods as well. The corrosion of arch wires can ultimately lead to mechanical failures and fractures in orthodontic materials [6]. The corrosion on the surface of NiTi arch wires may augment the mechanical friction appearing at the interface of the arch wire and bracket, which ultimately leads to decreasing action of free sliding during orthodontic treatments [13]. The main risk related to corrosion is the biological side effects upon the release of Ni [14]. Ni essentially leads to one of the most typical metal induced allergic mediated dermatitis in humans, causing more allergic effects as compared to the other metals [15].

Many researchers have studied the consequence of temperature variations on orthodontic alloy materials, and some of them have reported the negative effects of thermal processes on orthodontic treatments [15,16]. In fact, microstructural changes can occur due to these thermal stresses that essentially change the physical characteristics of the dental material [17]. Also, the rate of corrosion can drastically affect the mechanical strength of various NiTi arch wires with increasing cycles of thermocycling [18]. Another factor is acidic conditions or pH fluctuations that lead to the changes in the course of corrosion, where the food contents and acidic conditions are found to be very important factors [19]. Studies have reported that the corrosion process can be decreased in high pH conditions due to the alloy passivation originated from the protective film formation on the surface of alloy [20]. Hurst et al. [21] have earlier reported that the thermal transfer ribbon (TTR) should be higher than the oral temperature for application of shape memory phenomenon of NiTi alloys. Wataha et al. [22] demonstrated that briefly exposing Ni-based alloys to biologically meaningful levels of decreased pH can considerably enhance the amount of Ni released from the tested alloy. In comparison to Ni-based alloys, high-noble and noble alloys were resistant to this action and likely released fewer elements into the body locally and systemically. Any biological effects of the alloys may benefit from this reduced physical strain on the body.

The objective of this study is to evaluate the roughness of surface, load-deflection and mechanical characteristics of NiTi wires under different conditions such as change in pH and thermocycling processes. Also, a three-point bending analysis was employed to assess the load-deflection characteristics and surface roughness of the NiTi wires.

MATERIALS AND METHODS

Specimen preparation

The method of this study was *in vitro* or experimental. This study was approved by our Referee Committee for Thesis Defense. In this study, 40 rectangular section arch wires (NITI®, Ormco, USA) with 5-cm length were used. According to the manufacturers' indications, the wire dimensions are 0.016 inches.

The wire samples were divided into two groups, and each of them has 2 subgroups consisting of 10 samples. In first group, the samples were submerged in artificial saliva pH=7.4, which was further divided into two equal subgroups: 1-1 and 1-2. In second group, the samples were immersed in artificial saliva at pH=4.5, which was further divided into two equal subgroups: 2-1 and 2-2. The wires in the subgroups of 1-1 and 2-1 were not exposed to thermocycling, while the wires in the subgroups of 1-2 and 2-2 were exposed to thermocycling for 500 rounds in the temperature range from 5 to 55 ◦C in water. Artificial saliva was synthesized based on the protocol reported by Saghiri et al. [23]. The pH was fixed to be 4.5 via the addition of ascorbic acid. Before the evaluations, all the samples were ultra-sonically well-cleaned using ethanol for several minutes, rinsed in distilled-water and dried at 20-22 ◦C (68–72 ◦F).

Analysis of surface microstructure

Surface roughness parameters such as the ultimate tensile strength, stiffness, yield strength and surface roughness were calculated. The surface structure was investigated by a profilometer (RTD-200 Portable Surface Rough-ness tester, New Star, Australia) based on Hobbelink et al. [24] and Instron machine, respectively.

Assessment of Load-Deflection characteristics

After 30 days, the immersed arch-wires were taken out from the reaction solutions and investigated for their load-deflection characteristics at 37◦C in a water bath using the Universal Testometric Machine. In order to obtain the load-deflection curves, three copper (Cu) cylinders with a length of around 3 cm and a rectangular-cube bar with dimensions of 10 × 12 × 1 cm with fully smooth surface were developed. Two Cu-cylinders with central distance of around 14 mm were screwed tight, and the third Cu-cylinder was attached to the cross-head of the instrument. On each cylinder, a stainless-steel (SS) maxillary canine bracket was fixed with the slot-size of 0.018 inches and stabilized with cianoacrylate glue. The wire was then placed passively in the slot of the brackets such that the wire remained in the straight form, and then the wires were kept in the bracket slots and tied by elastomeric ligatures. The three-bracket bending tests were carried out on each sample. The instrument moved at a speed of 0.5 mm/min with the range of 2.4 mm, and the loading and unloading forces were documented. Finally, the load-deflection curves were plotted. The loading forces at 2.4-0.2 mm deflection (L2.4 to L0.2) and unloading forces at 2.2-0.2 mm with 0.2 mm deflection intervals (L2.2 to L0.2) were documented. The loading plateau (between L 0.6 and L 1.6), unloading (between UL0.6and UL1.6) and the difference between them (hysteresis) were determined for each sample.

Assessment of released ions

The sample solution was analyzed using the Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) to estimate the amount of Ni ions released. The ICP-MS was calibrated using the standard solution to detect Ni ions at a concentration of 100 and 10 ppb.

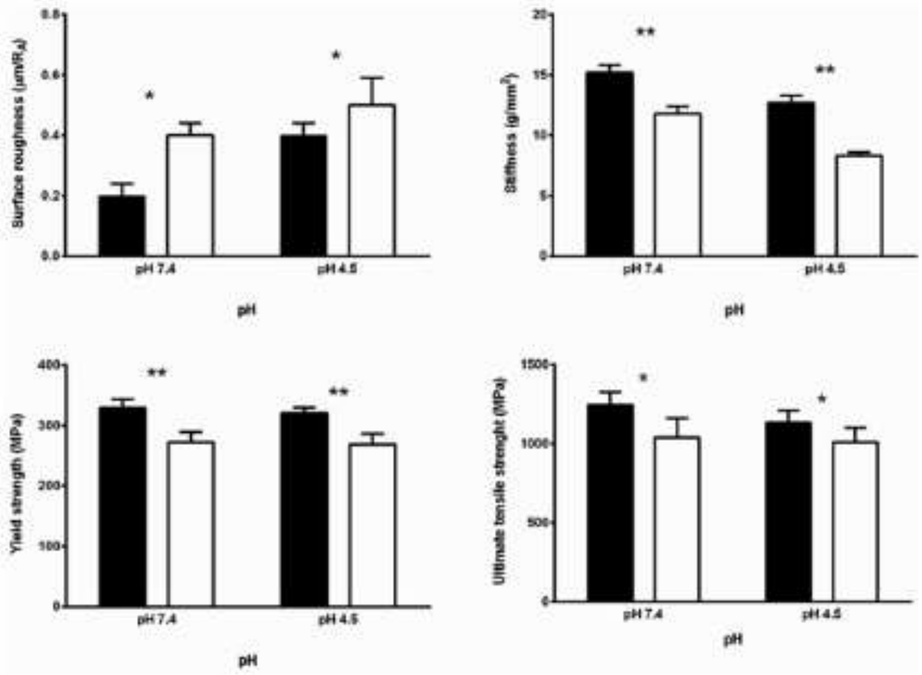
Statistical Analysis

Statistical analysis was carried out using professional software (SPSS V.22). All data-sets were presented as the mean-standard-deviation (SD). The Kolmogorov–Smirnov function was used to determine whether the data are normally distributed or not. Comparisons between the groups were performed using paired samples through Anova and following that by Post Hoc test.

RESULTS

The characteristics of NiTi arch wire samples under different conditions such as pH at 7.4 and 4.5 with and without thermocycling effects towards the bulk and surface mechanical properties such as stiffness, ultimate tensile strength, yield strength and surface roughness were evaluated, and the obtained results are shown in Table 1 and Figure 1. Similarly, Ni release from the NiTi arch wire under different pH and thermocycling conditions was investigated, and the obtained results are shown in Table 2.

Figure 1. Mechanical characteristics of NiTi archwire samples under different conditions.



Group 1: pH=7.4 with thermocycling; Group 2: pH=7.4 without thermocycling; Group 3: pH=4.5 with thermocycling; Group 4: pH=4.5 without thermocycling

Results showed that mechanical properties such as stiffness, ultimate tensile strength and yield strength were found to be the highest and the surface roughness was found to be the lowest at the pH of 7.4 without thermocycling as compared to that of the under thermocycling conditions. Similarly, under pH of 4.5, these mechanical properties of NiTi arch wires were found to be greater without thermocycling and surface roughness was found to be decreased as compared to that of the under thermocycling condition.

To further validate the surface compositional and structural instabilities in NiTi arch wires, the Ni release experiments were carried out under natural (7.4) and acidic (4.5) pH conditions with and without thermocycling. The obtained values are tabulated in Table 2. It can be observed that under the same pH value of 7.4, Ni release was found to be more under thermocycling condition. Similarly, irrespective of with and without thermocycling, the Ni-release was more under the acidic pH of 4.5. Overall, it is evident that the release of Ni was found to be the highest in case of pH 4.5 under thermocycling conditions. These observations signify that acidic and temperature conditions greatly influence surface chemical and structural properties of NiTi arch wires.

DISCUSSIONS

Our results showed that low pH (acidic condition) and presence of thermocycling lead to a significant increase in surface roughness and Ni release, and a significant decrease in stiffness, yield strength and ultimate tensile strength. The highest level of surface-roughness was observed in pH 4.5 under thermocycling, while the lowest surface-roughness was observed in pH 7.4 under non-thermocycling conditions. In a similar investigation, Yu et al. [4] reported that the surface roughness of NiTi wire was 0.63±0.07, which is higher than our results with thermocycling regardless of pH. This difference can be due to the use of different arch wires from different manufactures. Perinetti et al. [1] demonstrated that each NiTi arch wires showed different corrosion resistance under different conditions such as low pH, fluoride and thermocycling conditions. Further, their results also showed that thermocycling had no effects on the surface-corrosion of NiTi alloy arch-wires [1]. The results clarified that the thermocycling process considerably affects the mechanical properties of NiTi arch wires, and similarly, irrespective of with and without thermocycling, the mechanical properties were found to be improved at a pH of 7.4 as compared to a pH of 4.5. Natural pH facilitates the properties of NiTi arch wire to be more stable under non-thermocycling conditions. On the other hand, acidic pH and thermocycling considerably affected the stability of arch wires. This shows that both acidic nature and temperature strongly affect NiTi arch wires, where the decreased properties could be attributed to the formation of oxidation layers on the surface of NiTi wire. The stronger interaction under the given conditions can be clearly assessed from the observed reduced surface roughness of the samples. The impact of acidic conditions and thermocycling could be mainly attributed to the changes induced in NiTi arch wires' surface chemical composition and surface structural (morphology) properties. Such changes could have occurred through the surface etching process, leading to compositional and structural instabilities in the samples. In contrast, surface roughness in our study was much less. Ti alloys undergo surface oxidation before implantation, and its corrosion resistance and biocompatibility can be improved. Its mechanism involves the development of an oxidation protective layer on the surface of arch wire that cannot be dissolved in the presence of oxygen. In addition, the low pH can lead to the release of the oxidation layer of Ti and conversion of Tito TiO2and finally results in increasing the surface roughness of the material [25]. The thermocycling increases the layer separation process, which eventually increases the surface roughness as well [11,26]. The highest stiffness in our study was recorded at pH=7.4 without thermocycling. Previous studies have shown that the stiffness of NiTi arch wire changed after exposure to different acidity conditions [27], and thermocycling caused the metal particles to be closer together, which ultimately promoted the destruction of the metals and decreased the stiffness [27].

Another important observation of this present study is the amount of Ni release, which was directly dependent on the decrease in pH and presence of thermocycling (Table 2). Sifakakis and Bourauel [28] had performed *in vivo* histological and clinical analysis which indicated that the Ni release from arch wires, even in low-dose, leads to gingival overgrowth. In our study, the highest (6.23±1.20) and lowest amount (2.63±0.20) of Ni release was observed in pH4.5 with thermocycling and pH7.4 without thermocycling, respectively. Senkutvan et al. [29] found that there was no significant difference in Ni release between the stainless steel and NiTi wire. In accordance with these results, the average amount of daily Ni release is lower than the amount of daily intake of this ion. Thus, a systemic toxic effect from orthodontic appliances is highly unlikely [30]. The variations observed between different studies can be due to these reasons: differences in cross-sectional shape, diameter, and time of the experiment. Previous studies have shown that the amount of Ni release in NiTi wires is higher than other wires such as stainless steel. This issue can be for two reasons: formation of the titan–oxide layer and selective melting phenomenon [31].

In another study performed by Sheibaninia, the released Ni level was found to be similar to our results in the absence of thermocycling. In the presence of thermocycling, the released level of Ni was lower than the results reported by Sheibanini [10]. This observed difference may be because we have used 5 cm pieces of arch wire, but Sheibaninia used 5 cm of straight wire. Thus, pH and thermocycling can negatively influence Ni release from the wires, and these can be dominant factors in this process. This might have originated *via* the chemical etching of Ni ions from NiTi wires, where it causes chemical instabilities *via* the formation of oxide layers in NiTi on one level as well as structural instabilities *via* changing the surface morphologies in the system on another level. Both these influences over the chemical and physical properties of NiTi alloy can considerably reduce the overall performance of NiTi arch wires. Such phenomenon of Ni release-induced instabilities could be directly assigned to the observed changes in the mechanical properties of the NiTi system.

Furthermore, we have observed the highest yield strength and ultimate tensile strength at 7.4 pH under non-thermocycling condition and the lowest values at 4.5 pH in the presence of thermocycling (Table 2). However, higher yield strength and ultimate tensile strength were reported by Watacha et al. [32] and Gopalan et al. [33] respectively, as compared to the values obtained in our study. Such enhancements could be attributed to the difference in the cross-sectional dimension of the wires, environmental conditions and manufacturers.

However, our study had some limitations: (i) the simulated in vitro condition in this study could be different from the intrinsic in vivo conditions of the mouth; (ii) the pH and temperature in our study may be different from the real-time conditions (i.e., patient's mouth). However, all the analyses in this study were performed using the samples with suitable size and considered the effects of pH and thermocycling to study the mechanical and surface properties of NiTi wire.

CONCLUSIONS

Both acidic pH and thermocycling process considerably influence and negatively impact the properties of NiTi alloy arch wires. So, the acidic pH of 4.5 and thermocycling process considerably increase surface roughness of NiTi alloys, and decrease stiffness, ultimate tensile strength, and yield strength of the alloys. Also, these two influencing factors greatly increased Ni release, which validated the observation of decreased mechanical and surface properties. Ni release was essentially mediated by etching of Ni from the alloy that might have caused chemical instabilities while changing the surface structure (morphology) of the system.

Conflict of interest: The authors declare that they have no conflict of interest.

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Declaration of Interest statement: We declare that we have no conflict of interest.

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