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Research Report

The influence of artificial salivary pH on nickel ion release and the surface morphology of stainless steel bracket-nickel-titanium archwire combinations

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ABSTRACT

Background: *In the oral cavity, orthodontic appliances come into contact with saliva which may cause corrosion capable of changing their surface morphology due to the release of metal ions. Surface roughness can influence the effectiveness of tooth movement. One of the ions possibly released when body fluid comes into contact with brackets and archwire is nickel ion (Ni). Ni, one of the most popular components of orthodontic appliances, is, however, a toxic element that could potentially increase the likelihood of health problems such as allergic responses during treatment.* ***Purpose:*** *The purpose of this study was to investigate the effect of different artificial salivary pH on the ions released and the surface morphology of stainless steel (SS) brackets-nickel-titanium (NiTi) and archwire combinations.* ***Methods:*** *Brackets and archwires were analyzed by an Energy Dispersive X-Ray Detector System (EDX) to determine their composition, while NiTi archwire compound was examined by means of X-ray Diffraction (XRD). The immersion test was performed at artificial salivary pH levels of 4.2; 6.5; and 7.6 at 37°C for 28 days. Ni ion release measurement was performed using an Atomic Absorption Spectroscopy (AAS). Surface morphology was analyzed by means of a Scanning Electron Microscopy (SEM).* ***Results:*** *The chemical composition of all orthodontic appliances contained Ni element. In addition, XRD was depicted phases not only NiTi but also Ni, Titanium, Silicon and Zinc Oleate. The immersion test showed that the highest release of Ni ions occured at a pH of 4.2, with no significant difference at various levels of pH (p=.092). There were surface morphology changes in the orthodontic appliances. It was revealed that at a pH of 4.2, the surfaces of orthodontic appliances become unhomogenous and rough compared to those at other pH concentrations.* ***Conclusion:*** *The reduction of pH in the artificial saliva increases the amount of released Ni ions, as well as causing changes to the surface morphology of brackets and archwires.*

Keywords: *salivary pH; Ni ions release; NiTi archwires; SS brackets*

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INTRODUCTION

In the present day, the demand for orthodontic treatment is increasing.[1] Two main components are frequently employed in this form of dental treatment: brackets and archwire. The most common bracket material is stainless steel (SS) because of its affordability and appropriate physical properties. The generally-used archwire material is nickel titanium (NiTi) due to its favorable results with regard to tooth movement.[2]

In the oral cavity, orthodontic appliances invariably come into contact with saliva which produces a electrochemical reaction commonly referred to as corrosion.[3] This process has the potential to change the chemical structure of orthodontic appliances and can only be measured by analyzing the ions released.[4] One of the ions which can potentially be released and often causes allergic reactions is nickel (Ni).[5,6]

The potential negative health effects resulting from exposure to Ni and its compounds have been investigated

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for over a century, with it being established that Ni can induce hypersensitivity, dermatitis, and asthma.[7] In addition, Ni can cause DNA damage to oral mucosa, suppression of chemotaxis of leukocytes, and changes in enzyme activity.[8,9] Ni is known to be an indispensable element for both human beings and animals. In general, the most significant exposure for humans occurs through diet, air, drinking water, and jewellery with the average dietary intake of Ni estimated to be 200-300 μg/day.[10]

There are several factors potentially affecting the release of metal ions from orthodontic appliances, including; alloy composition, salivary pH, and duration of use.[9] In this study, an evaluation of the alloy composition was conducted by means of an energy dispersive x-ray detector system (EDX) and x-ray diffraction (XRD) in order to identify their compounds. The effect of salivary pH on the Ni ions released was analyzed using an atomic absorption spectroscopy (AAS). The release of ions can also be observed by examining the surface morphology of orthodontic appliances.[11] That of a stainless steel brackets-NiTi archwire combination was analyzed by means of a scanning electron microscopy (SEM). This in vitro study was performed through a classic procedure involving the immersion of the sample in artificial saliva at various pH values over an extended time period. The purpose of this investigation was to examine the influence of salivary pH on the release of Ni ions and surface morphology changes in SS brackets-NiTi archwire combinations.

MATERIALS AND METHODS

The artificial saliva employed consisted of Fusuyama Meyer artificial saliva (Morvabon, Tehran, Iran) with the following chemical composition: 0.4 gr NaCl; 0.4 gr KCl; 0.795 gr $CaCl_2.H_2O$; 0.78 gr $NaH_2PO_4.H_2O$; 0.005 gr $Na_2S.9H_2O$; 1 gr Urea and 1000 ml distilled water at 37 ± 1°C. The artificial saliva solution was adjusted with a small amount of HCl and measured potentiometrically using a pH meter (ebro® Electronic PTH 810, Germany) to provide a certain level of pH. The levels of salivary pH at 4.2, 6.5, and 7.6 simulated conditions within the oral cavity and were monitored at days 14 and 28, thereby simulating the time interval of the appliances fitted in the oral cavity. The pH levels of 6.5 and 7.6 were within the range of naturally-occurring human salivary pH, whereas a pH of 4.2 was intended to simulate acidic conditions that occasionally occurs when one eats or drinks acidic foods or beverages (e.g. lemon, fruit juice, Coca-Cola).[7]

Brackets (Protect, China) and archwires (Protect, China) were analyzed using SEM (ESEM Quanta 400 FEG, FEI, Japan) equipped with EDX (EDAX TEAM™, AMETEK, JAPAN) and XRD (Shimadzu XRD-6000 diffractometer, Columbia, USA). An EDX uses the x-ray spectrum emitted by an SS bracket or NiTi archwire to subsequently identify the specific energy of characteristic x-ray peaks of each element. XRD was used to confirm characterization of the material content by analyzing the crystal structure and to compare the results with those contained in a component structures database as a means of determining the compound of the NiTi archwire.[11]

This study simulated fixed orthodontics treatment of half of the maxillary and mandibulary arches (from central incisor to first molar). All orthodontic materials used were produced by Zhejiang protect medical equipment (China). The bracket meshes were coated with adhesive resin (Xeno ortho paste, Japan) to protect them from corrosion.

Three groups represented the focus of this study and were divided into two control groups (bracket and archwire only) and one treatment group (bracket-wire combination). The bracket group consisted of 5 brackets and 1 buccal tube per jaw immersed in artificial saliva at a pH level of 6. The archwires group was composed of 6 cm of 0.021 x 0.025-inch wire for the upper and lower jaws each of which was immersed in artificial saliva at respective pH levels of 4.2 and 6.5, whereas the treatment group consisted of a half set of a bracket-archwire combination for both the upper and lower jaws. Each 6 cm length of 0.021 x 0.025-inch wire was ligated by means of elastic in the bracket slot and immersed in the artificial saliva at pH levels of 4.2, 6.5, and 7.6. Each group had 3 samples for its repetition, which were 3 samples for bracket groups, 6 samples for archwire groups (3 samples respectively for pH 4.2 and 6.5), and 9 samples for treatment groups (3 samples respectively for pH 4.2, 6.5, and 7.6). Each sample group was immersed in 180 ml artificial saliva. 10 ml sample of artificial saliva was collected at days 14 and 28, with the release of Ni ions in this sample subsequently being analyzed by AAS.

On day 28, the brackets and wires were removed from the artificial saliva and their respective surface morphologies were subjected to SEM analysis. The statistics used for data analysis on a sample resulted from a paired t-test to compare the release of nickel ions during immersion on days 14 and 28. One-way analysis of variance (ANOVA) was conducted to compare the amount of nickel ions released, while a Kruskal Wallis test was conducted for morphologic surface comparison in the tested pH/groups.

RESULTS

EDX analysis showed that the elemental compositions of the SS brackets were C, O, F, Al, Si, Cr, Fe, and Ni (Figure 1), whereas those of NiTi wires were C, O, Zn, Al, Si, S, Cl, Ti and Ni (Figure 2). The XRD analysis showed that NiTi archwire contains other compounds besides NiTi such as nickel titanium silicon and zinc oleate (Figure 3). In addition, the AAS analysis confirmed that the highest release of Ni ions occurred at a pH of 4.2 in the artificial saliva immersion, while the lowest was at a pH of 7.6 (Table 1).

One-way Anova statistical analysis confirmed there to be no significant difference in the number of Ni ions released at various pH levels (p=.092). When evaluating

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the effect of time on Ni ion release, each treatment group was revealed to increase the volume of nickel ions released across the 14-day and 28-day time intervals, but no statistically significant difference existed (p=.055) (Table 1).

The SEM images showed the surface morphology of brackets and archwires were unhomogeneous and rough due to contact with artificial saliva. The image at a pH of 4.2 revealed rougher scratches than the others (Figures 4, 5, 6, 7).

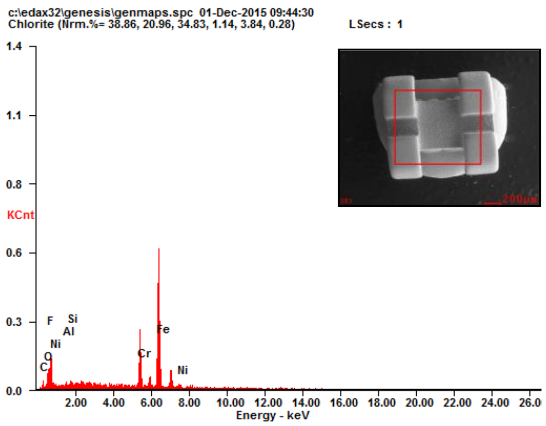

Figure 1. EDX analysis for SS bracket.

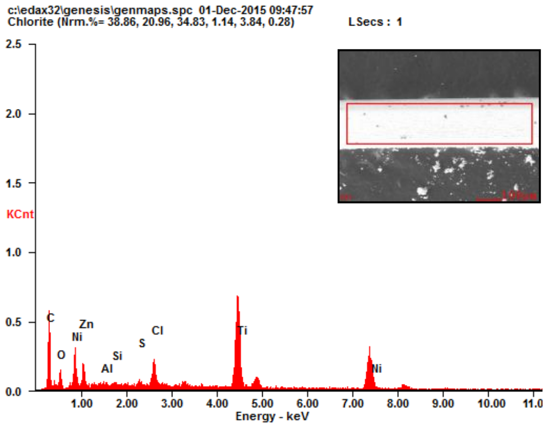

Figure 2. EDX analysis for NiTi archwire.

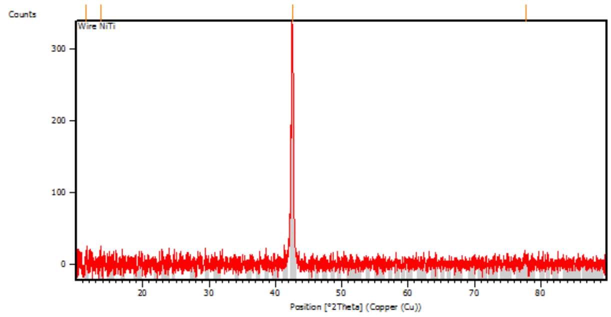

Figure 3. XRD analysis for NiTi archwire.

Table 1. The amount of nickel ions released

pH	Ni ions release (ppm)					
	SS bracket attached NiTi Archwire		Control sample			
			Group Archwire		Group Bracket (10 brackets+2 *buccal tubes*)	
	14 days	28 days	14 days	28 days	14 days	28 days
4.2	0.7995	0.9118	0.2596	0.2669	-	-
6.5	0.5580	0.6848	0.0000	0.0000	0.5157	0.8309
7.6	0.1937	0.3974	-	-	-	-

Table 2. The comparison between present study and the other studies

References	Quantity			*Part of Whole Appliance*	Materials		Ni ion release		Time (days)
	Bracket	*Band/ Tube*	*Wire*		Bracket	*Wire*	Acid pH	Normal pH	
Present Study	10 (Protect)	2 (Protect)	½ UA, ½ LA (Protect)	½	SS	NiTi	0.9118 ppm (pH=4.2)	0.6848 ppm (pH=6.5)	28
Kuhta *et al* (2009)	5 (Dentaurum)	1 (Dentaurum)	½ UA (Dentaurum)	¼	SS	NiTi	5430.76 ppb (pH=3.5)	166.88 ppb (pH=6.75)	28
Mikulewicz *et al* (2012)	20 (3M)	4 (3M)	2 (AO)	1	SS	SS	-	573 µg/L (pH=7)	30

* UA=upper arch; LA=lower arch; pH=potential of hydrogen; Ppb=part per billion; Ppm= part per million; and AO, American Orthodontics.

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DISCUSSION

There are many aspects of long-term orthodontic treatment to be considered, including the materials used. Metal alloys are the most common whose microstructure, due to the manufacturing process, influences their mechanical properties and corrosion behaviour. This can be a process involving casting, machine-based milling, or metallurgical powder.[4]

The pH level of the solution can accelerate the corrosion process, as confirmed by the release of metal ions which may, in turn, cause changes in the metal's surface. The aforementioned corrosion process will release metal ions directly into the solution or damage the protective layer on the metal surface resulting in the release of ions. This process is continuous and cumulative, invisible to the human eye and only measurable by the ions released.[4] In this study, the Ni ions were released directly from the NiTi archwire because it contained no TiO_2 substances acting as a protective layer such as are generally provided in NiTi alloys as reported in the XRD results (Figure 3).[12]

Several previous studies have shown that metal ions can be released by most orthodontic appliances, potentially leading to an inflammatory reaction, irritation or dermatitis.[4,13,14] Small concentrations of metal ions could affect the mucous membrane cells. It has also been reported that the nickel ions released can accumulate in the cells over time and may endanger them by, for example,

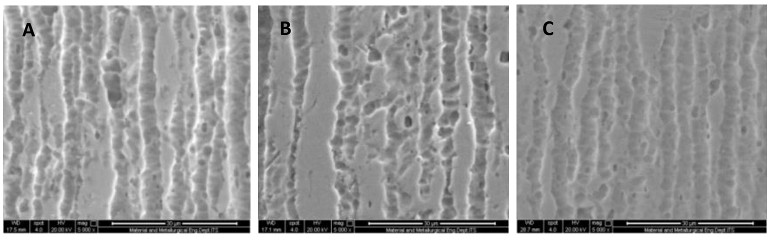

Figure 4. The surface morphology of NiTi archwires in the treatment group after 28 days' artificial saliva immersion at pH of: A) 4.2; B) 6.5; and C) 7.6. (5000x magnification).

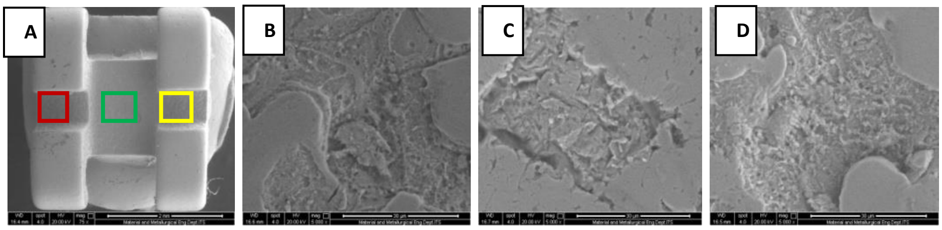

Figure 5. The micromorphologic characteristic of SS brackets slot surfaces in treatment group after 28 days artificial saliva immersion at pH of 4.2 with various magnification. **A**. 75x, **B**. □ 5000x, **C**. □ 5000x, **D**. □ 5000x.

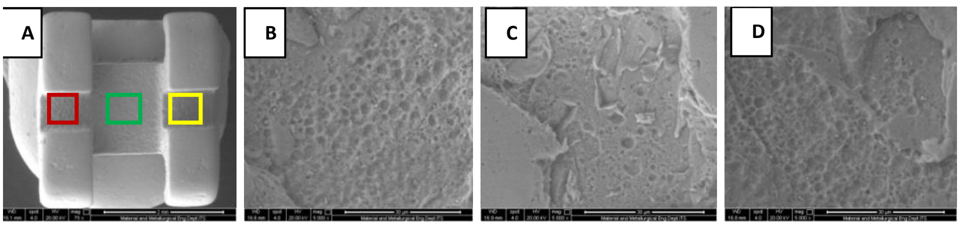

Figure 6. The micromorphologic characteristic of SS brackets slot surfaces in treatment group after 28 days artificial saliva immersion at pH of 6.5 with various magnification. **A**. 75x, **B**. □ 5000x, **C**. □ 5000x, **D**. □ 5000x.

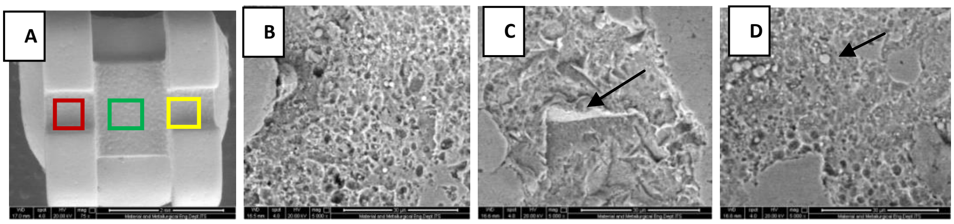

Figure 7. The micromorphologic characteristic of SS brackets slot surfaces in treatment group after 28 days artificial saliva immersion at pH of 7.6 with various magnification. **A**. 75x, **B**. □ 5000x, **C**. □ 5000x, **D**. □ 5000x.
(→: saliva deposit)

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altering the chemotaxis of leukocytes and the synthesis of DNA and enzyme activities.[8,9] Ni solution (0.05mol/L) may impede polymorphonuclear leukocyte phagocytosis, impair leukocyte chemotaxis, and stimulate neutrophils to become aspherical and move more slowly.

According to EDX analysis, both SS brackets and NiTi archwires contain the Ni element. The chemical bonding of nickel atoms to inter-metallic compounds is weak, causing an increase in the release of Ni ions from alloy which may subsequently compromise the biocompatibility of appliances.[16] In addition, during the present study, the release of Ni ions was also shown to be influenced by environmental factors, such as salivary pH (replicating oral conditions) and the duration of immersion (simulating the time interval of the appliance in the mouth).[9] The measurement of Ni ion release in this group revealed that the highest concentration occured in the artificial saliva immersion at a pH of 4.2 (0.9118 ppm/28 days), with the lowest at a pH of 7.6 (0.3974 ppm/28 days). Meanwhile, the Ni ion release at a pH of 6.5 was 0.6848 ppm. These values are comparable to those obtained by Kuhta *et al*. and Mikulewicz *et al*.[9,17] Kuhta *et al*. simulated the orthodontic appliances which consist of incisor to premolar maxillary brackets (Dentaurum, Germany), molar bands and ligature wire (Dentaurum, Germany), NiTi archwire 0.016 x 0.022 over 28 days of observation.[9] The research showed that nickel ion release at pH = 6.75 was 166.88 ppb (0.1669 ppm), whereas at pH = 3.5 it was 5430.76 ppb (5.4308 ppm). A similar study was undertaken by Mikulewicz *et al*. using neutral pH=7 and simulated orthodontics appliances with 20 SS brackets (3M Unitek, USA), 4 bands (3M Unitek, USA), two 0.017" x 0.025" SS archwires (American Orthodontics, USA) and 20 metal ligatures (American Orthodontics, USA). The study showed that Ni ion release was 573 μg/L (0.573 ppm) over 30 days.[17] It can be concluded from the present study that the orthodontic appliances incorporating SS brackets-NiTi archwire combinations were acceptable because the concentration of nickel ions released in acidic condition was not as high as in those referred to above (Table 2).

The effect of salivary pH on the release of Ni ions can be observed in the surface morphological of SS brackets-NiTi archwire combinations after immersion in the artificial saliva for 28 days. The surface morphology of NiTi archwire at a pH of 4.2 appeared to be rougher than that of one at 7.6. This finding is in line with the previous study which concluded that the higher the acidity, the rougher the surface of the NiTi archwire. This condition relates to the volume of Ni ions released. Under acidic conditions, this increases with the morphology of the surface, consequently, appearing to be rougher.[18]

To confirm the occurrence of Ni ion release during this research, two control groups (bracket group and archwire group) with the artificial saliva immmersion at pH of 6.5 (Table 2) were analysed. Additional investigation of the NiTi archwire group was conducted at a pH of 4.2 because no Ni ion release was detectable at one of 6.5, whereas at a pH of 4.2 the figure was 0.2669 ppm at 28 days. These results indicate that NiTi archwire (Protect, China) was acceptable at a pH of 6.5 even though the release of Ni ions can still occur on the wire by changing the salivary pH to be more acidic.

The level of nickel ion release of the SS bracket-NiTi archwire combination (the upper and lower jaws) at various pH levels over 28 days remained below the toxic level of leukocyte chemotaxis (2,5 ppm).[19] This value is still tolerable for human beings because it is below the toxicity level of nickel (30 ppm).[13] As a consequence, this orthodontic appliance (Protec, China) is safe for use over a period of 28 days. However, further research of greater duration is needed in order to determine the exact period during which the orthodontic appliance can be safely used. In conclusion, decrease in salivary pH increases the corrosion process, thereby releasing Ni ions from the metal alloys contained in appliances and affecting the homogeneity of the surface morphology of the SS bracket-NiTi archwire combination.

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