Original Article

Nickel Content of As-Received and Retrieved NiTi and Stainless Steel Archwires: Assessing the Nickel Release Hypothesis

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Abstract: This study assesses the nickel content of as-received and retrieved stainless steel and NiTi archwires alloys. New and used brand-matched, composition-matched, and cross section–matched archwires were subjected to scanning electron microscopy and energy-dispersive electron probe microanalysis. Elemental analysis was performed on three randomly selected areas, and the nickel content, expressed as ratios of Ni/Ti (in NiTi wires) or Ni/Fe (in stainless steel), was statistically analyzed with a *t*-test ($\alpha = .05$). No changes were detected with respect to Ni content ratios between as-received and retrieved NiTi or stainless steel wires, suggesting an absence of nickel release. Wear and delamination phenomena on the wire surface and the formation of galvanic couple between the stainless steel wires and bracket brazing materials intraorally may modify the corrosion susceptibility of the wire alloys in clinical conditions. (*Angle Orthod* 2004;74:151–154.)

Key Words: Nickel release; NiTi; Stainless steel archwires; EDS

INTRODUCTION

The fate of particulate masses derived from biomaterials in contact with biological fluids or tissues has long been considered a critical issue determining the biological properties of biomaterials.[1,2] The particular emphasis recently placed on Ni arises from the abundance of evidence connecting this element with a wide range of pathological conditions.[3–5] Specifically, Ni amounts as low as 2.5 ng/mL (ppm) have been found to impair the chemotaxis of leukocytes and stimulate neutrophils to become aspherical and move slowly.[6] In addition, Ni at concentrations in the range of those found to be released from dental alloys has been shown to activate monocytes and endothelial cells and affect the expression of intercellular adhesion molecule 1 by endothelial cells.[7] Most importantly, nontoxic concentrations of Ni may inflict direct DNA base damage and site-specific DNA strand scission (single-strand breaks),[8] whereas an indirect route of nickel-induced DNA alteration involves the inhibition of enzymes known to restore DNA breaks.[9] Lastly, Ni ions at nontoxic concentrations may promote microsatellite mutations and inhibit the repair of nucleotide excisions, thereby contributing to genetic instability.[10,11]

The American Iron and Steel Institute type 316 L austenitic stainless steel alloy (''L'' denotes low carbon content), which is primarily used for biomaterials manufacturing including stainless steel brackets and archwires, contains Ni in the range of 10–14% (wt%), whereas the Ni content of NiTi wire alloys may exceed 45%.[12] Release rates have been frequently cited in the literature through in vitro methodological approaches.[13]

However, in vitro studies encompass problems pertinent to the lack of clinical relevance of the experimental conditions, incorporation of assumptions concerning the kinetics of release, and adoption of simplifications in simulating the clinical milieu.[14] Further, animal models may be of questionable reliability owing to the species-specific variations of metabolic and excretory potentials for Ni.[15] Lastly, the investigation of metal content in human biological flu-

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ids, such as saliva or blood, is complicated by the high individual variability and the inability to derive an estimation of the cumulative release of metals over the full term of treatment.[14]

Although the compositional alterations accompanying the intraoral use of stainless steel brackets has been demonstrated previously,[16] the implication of stainless steel and NiTi archwires in the issue of Ni release from orthodontic alloys remains unknown.

The hypothesis tested in this investigation was that the intraoral exposure of stainless steel and NiTi orthodontic archwire alloys induces Ni release, identified by a decrease in the Ni content of retrieved materials relative to their as-received counterparts. This study assesses the Ni content of unused and retrieved stainless steel and NiTi archwires.

MATERIALS AND METHODS

The NiTi and arch-formed stainless steel wires included in this study (German Orthodontics, San Jose, Calif) were retrieved during the routine visits of orthodontic patients. Each wire specimen was placed in a self-closed, sterilizing plastic bag, and the following information was recorded: (1) composition of the alloy, (2) date of archwire placement, (3) dental arch of insertion, and (4) date of archwire removal. All archwires collected were of rectangular 0.016 $\times$ 0.022-inch (0.41 $\times$ 0.56 mm) cross section and had been ligated to stainless steel brackets (Forestadent, Pforzheim, Germany) of 0.018-inch (0.45 mm) slot size by means of elastic modules or stainless steel ligatures. The archwire retrieval procedure yielded 45 retrieved wires (20 stainless steel and 25 NiTi) with a mean intraoral service period of four months (range, 1.5–12 months). The collected archwires were rinsed with double-distilled water to detach any loosely bound deposits, and specimens of 10 mm length were prepared. Identical specimen treatment was followed for unused archwires from the manufacturer by matching the composition and size with those of the retrieved materials.

Scanning electron microscopy and energy-dispersive X-ray microanalysis (SEM-EDS) were used to assess the elemental composition of the wires after intraoral exposure relative to the as-received condition. For this purpose, wire segments were bonded to aluminum stabs, vacuum coated with a thin layer of conductive carbon, and examined under an SEM unit (Quanta 200, FEI, Hillsboro, Ore.) equipped with a Si (Li) EDS (CDU sapphire NEW XL 30 EDAX, Mahwah, NJ) with super ultrathin window (Be). Spectra were obtained at three randomly selected regions on the surface of the wires under the following conditions: 25 kV accelerating voltage, 100 μA beam current, 500$\times$ original magnification with a 0.26 $\times$ 0.26-mm sampling window, 100 seconds acquisition time, and 30–40% dead time. The quantitative analysis of the percent weight concentration of the probed elements was performed by nonstandard analysis (by using ZAF correction procedures) with a $\pm$3% accuracy of the estimate values. For standardization purposes, the Fe content was used as an internal standard in the stainless steel wires, and nickel content was expressed as a nickel/iron ratio. The choice of Fe as a standard is based on its stability in the alloy microstructure, which derives from its dominant proportion relative to other elements (Fe content in 316 L stainless steel type exceeds 60% weight). Similarly, the nickel content of the NiTi wires was expressed as a nickel/titanium ratio.

Ni content values of as-received and retrieved specimens for stainless steel and NiTi wires were statistically analyzed with the *t*-test at the $\alpha = .05$ level of significance.[17]

TABLE 1. Ni/Ti Ratios Obtained for the Two Sampling Conditions (as received and retrieved) of the NiTi Archwires Included in the Study

	Ni/Ti Content Ratio	
NiTi Archwires	Mean	SD
As received	1.227	0.010*
Retrieved	1.228	0.005*

* Not significantly different at the $\alpha = .05$ level.

TABLE 2. Ni/Fe Ratios Obtained for the Two Sampling Conditions (as received and retrieved) of the Stainless Steel Archwires Included in the Study

Stainless Steel Archwires	Ni/Fe Content Ratio	
	Mean	SD
As received	0.125	0.003*
Retrieved	0.124	0.003*

* Not significantly different at the $\alpha = .05$ level.

RESULTS

In Table 1 the Ni/Ti content ratios for NiTi wires are shown. No statistical significant difference was identified between the as-received and retrieved states. Table 2 demonstrates the Ni content ratios of the stainless steel wire alloy, indicating a lack of difference and implying that no release occurred in vivo.

DISCUSSION

Retrieval analyses have gained interest in biomaterials research because of the critical information derived from investigating the performance of the material in the environment in which it was intended to function. The main factor that distinguishes the in vivo environment from various storage media is the presence of complex oral flora and their by-products, which along with local microenvironmental variables, may form specific conditions, which cannot be simulated under current in vitro research methodological approaches.[18] Retrieval approaches have been widely used in medicine and dentistry, leading to the for-

mulation of guidelines for handling and processing of retrieved materials by international organizations.[19]

The recognized hazardous action of nickel has stimulated the European Union legislation to enforce a regulation limiting the Ni release from utensil materials and jewelry to 0.5 μg/cm^2 of material surface per week for at least two years.[20] Nonetheless, ionic nickel is an important nutrient factor as evidenced by its incorporation in dietary supplements in the order of five μg/capsule for one capsule daily regimen.[21] The average person receives amounts of nickel in the order of 170 μg through ingestion and 0.4 μg through inhalation.[22] The foregoing discussion indicates that the bonding state and composition of nickel compounds may modulate its biological properties.

Research on the intraoral alterations of orthodontic archwires has revealed a wide array of degradation phenomena.[18] The enhanced deterioration of the wire surface coming in contact with the bracket slot wall has been illustrated and explained on the basis of development of compressive forces accompanying wire activation through ligation and possible frictional damage produced inside the slot. Further aging may be induced by treatment procedures such as sliding mechanics and the associated contact of bracket slot walls with the wire surface. Specifically, recent studies have suggested that some notching occurs on the wire surface during treatment owing to masticatory loads. This effect has a wide range of severity depending on the bracket-archwire combination, with the ceramic brackets imposing the most pronounced alterations on the wire surface.[23] It is worth noting that the foregoing evidence has not been taken into consideration in several in vitro approaches typically used to simulate the intraoral environment, thus precluding the extrapolation of clinically meaningful conclusions. Currently, there is a lack of evidence about the contributory role of intraoral aging on the extent and kinetics of nickel release.[16]

In general, release of elements from alloys may be enhanced by corrosive processes.[24] The anticorrosive properties of stainless steel and NiTi archwires is because of the formation of oxide layers on the surface of the alloys, which inhibit further corrosion. The passive film on stainless steel is composed mainly of Cr oxide compounds, with smaller amounts of Fe, Ni, and Mo. On Ti alloys several oxides are formed upon exposure to air, including TiO_2, TiO, and Ti_2O_5, with TiO_2 being the most dominant.[14] The Ti oxide layer formed on titanium alloys such as the NiTi wires is more stable than its chromium counterpart, particularly in environments containing chloride anions.[24] The formation of oxide layer may inhibit the outward movement of ions, thereby acting as an obstacle for release.

The results of this study should not be viewed as a conclusive evidence of the lack of ionic release from alloys. Apart from ionic release, leaching of corrosive and wear products in the oral cavity may be of critical importance for the biologic performance of materials. Wear-induced delamination may not be detected by spectroscopic techniques, which assess compositional changes, because the loss of macroparticles does not alter the overall elemental composition of the material. The intraoral aging of archwire alloys has been shown to include delamination processes with loss of wire fragments, which arises probably from wear between the wire surface and the slot walls.[18] Wear induced by the contact of materials with tissues, fluids, or other biomaterials may alter the biologic performance of a material[25] by producing wear particles, thereby increasing the effective contact surface with the tissue, which elicits a tissue reaction. Alternatively, adhesive wear may be generated by sliding of heavy rectangular wire segments onto the bracket slot walls. The implication of wear in the variation of the bracket slot–archwire friction has been shown by Kusy and Whitley,[26] who proposed that the formation of particles due to adhesive wear may adversely affect the velocity of movement.

Much research in the broader biomedical materials literature has dealt with the implication of wear-derived debris in the biological properties of the material.[27,28] The significance of this parameter arises from the association of particle size with the uptake capacity of phagocytes. Animal studies have indicated that although foreign body particles with a diameter of 0.080 μm may be phagocytized at an extent of 24,000 particles/cell, an increase in the size of the particulates by 38 times to three μm results in a 8000-fold decrease in the number of particles phagocytized per cell.[29] This exponential reduction in the phagocytic capacity of cells is indicative of an impaired material particle uptake and transport; thus, large particles may not be efficiently handled by phagocytosis. The foregoing may be of interest to orthodontics considering that the particulate irregular-shaped masses delaminated from the wire surface may reach lengths as high as 30 μm.[18]

Lastly, the ligation of wires in stainless steel brackets intraorally may result in the generation of a galvanic couple between the stainless steel wire and the components of the bracket. It has been demonstrated that manufacturing of the stainless steel brackets involves two different stainless steel types: a lower-modulus (such as the 316 L type) type used for the fabrication of the base providing efficient debonding through a peel-off effect and a higher-modulus type (such as the S17400 precipitation hardening) used in wing manufacturing, which facilitates the stiffness required to resist plastic deformation during load application.[30] These alloys are joint together with brazing materials mainly consisting of silver, nickel, or gold.[31] Therefore, the contact of more precious elements with those present in stainless steel alloys may have an unpredictable outcome on the corrosion susceptibility of the wire alloys in vivo.

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