



Evaluation of Coating Stability, Surface Characteristics and Biocompatibility of Nano Particle Coated Stainless Steel, Nickel-Titanium and Beta-Titanium Orthodontic Arch Wires in Comparison with Uncoated Wires: An In-Vitro Study

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Received 2018 April 20; Revised 2018 October 12; Accepted 2019 February 12.

Abstract

Background: Friction plays a major role during tooth movement as it takes up to 60% of the applied force, hence reducing the force available for tooth movement. Coating the surface of orthodontic wires by various techniques is being developed to improve their mechanical and biological properties.

Objectives: To evaluate the characteristics of nano particle coated and uncoated stainless steel, nickel-titanium, and beta-titanium wires for (1) coating stability, (2) surface characteristics and (3) biocompatibility after 21 days of exposure to artificial saliva.

Methods: Six types of wires were tested for coating stability, surface characteristics before and after exposure to artificial saliva using scanning electron microscope and the artificial saliva was tested for leaching of ions by inductively coupled plasma optical emission spectrometer.

Results: Coating thickness was reduced in each group after exposure to artificial saliva which was statistically significant. Significant changes in surface morphology such as delamination and irregularity of the coating was noted. Coated NiTi wires showed lesser leaching of ions when compared with uncoated NiTi wires but the difference was not statistically significant.

Conclusions: Coating delamination and irregularities were seen in many areas. The highest concentration of ions leached among all the groups were of iron, followed by silica.

Keywords: Coated Archwires, SEM, Nano Particles, Coating Stability, Surface Roughness

1. Background

Metal alloys of various types are routinely used in dentistry. In 1930s and 1940s gold was used for the fabrication of accessories in orthodontics. The advent of stainless steel replaced gold and was used for the fabrication of orthodontic bands, brackets and wires. In the 1970s, Nickel-Titanium alloys were introduced for use as orthodontic wires and are now used frequently, especially in the initial stages of treatment with fixed appliances, because of their favorable mechanical properties (1, 2).

Nickel is an identified allergen which maybe released during nickel-titanium and stainless steel archwire use. According to previous research patients wearing orthodontic appliances showed high concentrations of nickel in the saliva and oral mucosa (3). Burning sensation, gingival hyperplasia, angular cheilitis, erythema multiforme, stom-

atitis, perioral rash, and loss of taste are some oral signs and symptoms of nickel allergy (3). Fixed appliances also provide an ideal environment for colonization of microorganisms since orthodontic appliances can be difficult for patients to clean and maintain adequate oral hygiene (4). Concomitant increases in the prevalence of nickel hypersensitivity during the last decade have created mounting interest in alloy compositions and the release of metal ions during treatment, and their side effects.

Friction plays a major role during alignment and leveling and also during space closure. If up to 60% of the active force is dissipated as friction it reduces the force available for tooth movement such that an additional force must be applied in order to overcome the frictional force. The problems related to increased friction during orthodontic treatment may range from difficulty in anchorage control, increase in duration of orthodontic treatment and in-

crease in the risk of root resorption (5).

Attempts to reduce friction and improve both, the mechanical and biological properties of orthodontic wires, a few strategies were developed such as alteration of the bracket design, use of different types of alloys, surface treatment or coating of wires using various techniques and materials, as well as modifying the surface of wires and brackets which were successful to some extent (5).

Nanoparticles of metal dichalcogenide with fullerene-like structure were discovered in the early 1990's and are known to serve as excellent solid lubricants under various conditions. This can be used for coating the orthodontic wires or brackets in order to reduce the friction during the orthodontic treatment (6, 7).

2. Objectives

Synthesis of Nano Clear has allowed remarkable improvement of wear and friction properties due to the penetration of the Nano Clear nanoparticles into the interface between wire surfaces.

Hence this study was undertaken to evaluate the material characteristics of nano particle coated and uncoated stainless steel, nickel-titanium, and beta-titanium for the parameters (1) coating stability, (2) surface characteristics and (3) biocompatibility (release of nickel, chromium, silica and iron from the wires) after 21 days of exposure to artificial saliva.

3. Methods

The materials used were:

1. Ormco, USA - $0.019 \times 0.025''$ beta titanium and stainless steel straight length wires;
2. G and H Wire Company, USA - $0.019 \times 0.025''$ NiTi straight length wires;
3. Nanoparticles coating precursor solution was collected from progressive chemicals Pvt. Ltd;
4. The artificial saliva used in this present study was prepared according to Macknight-Hans and Whitford formula (8).

Experimental materials comprised of six types of wires, classified into six major groups, each group consisted of 15 samples which were tested, therefore 90 wire samples were tested, for coating stability, surface characteristics before and after exposure to artificial saliva and the artificial saliva itself was tested for leaching of ions such as nickel, chromium, iron and silica (Table 1).

Table 1. Experimental Materials

Group	Wires Used in the Group
Group I	Nanoparticle coated NiTi wires ($0.019 \times 0.025''$)
Group II	Nanoparticle coated beta titanium wires ($0.019 \times 0.025''$)
Group III	Nanoparticle coated stainless steel wires ($0.019 \times 0.025''$)
Group IV	Commercially available uncoated NiTi wires ($0.019 \times 0.025''$)
Group V	Commercially available uncoated beta titanium wires ($0.019 \times 0.025''$)
Group VI	Commercially available uncoated stainless steel wires ($0.019 \times 0.025''$)

3.1. Coating of Wires

Fifteen wire samples from each of the groups I, II and III were coated with nanoparticle film using 100 mL Nano Clear. The wires were first held under running water to discard any dust particles and later cleaned with a degreasing powder in order to remove any interference with the coating procedure. Nanoparticle coating solution was prepared by mixing Nano Clear (Figure 1) along with the Nano Clear solvent (Figure 2) in a ratio of 1:1. The orthodontic wires were kept dipped into the nanoparticle solution for 30 minutes (Figure 3). Later the wires were removed and kept in a hanger where they were further painted with nanoparticle solution. The wires were then air dried for 2 minutes. The wire with the hanger was then placed in the hot air oven at 160°C for 3 minutes. In this way the commercially available orthodontic wires were coated with nanoparticles.

Once the coating on the wire samples was achieved, they were observed under the scanning electron microscope (F E I Quanta FEG 200) along with the uncoated wire samples from group IV,V and VI at 100X, 200X, 500X, 1000X and 5000X magnification to assess their surface characteristics. At 1000X magnification, measurements were made of the coating thickness (Figures 4-6). The coated wire samples were then placed in test tubes filled with artificial saliva for 21 days, after which the wires were removed and the coating thickness was remeasured. The artificial saliva was analysed using inductively coupled plasma optical emission spectrometer (iCAP 6000 series ICP by thermo scientific) for the release of nickel, chromium, iron and silica.

3.2. Statistical Analysis

Minitab (www.minitab.com) was used to analyze the data. Two ways analysis of variance of means was used to assess the surface roughness results of each group to verify the influence of coating and of exposure to artificial saliva and the interaction of both factors on the results.



Figure 1. Nano Clear



Figure 2. Nano Clear solvent

4. Results

4.1. Evaluation of Coating Thickness

Pre-exposure coating thickness for NiTi, beta titanium and stainless steel wires with minimum, maximum and



Figure 3. The wires placed in the coating solution for 30 minutes

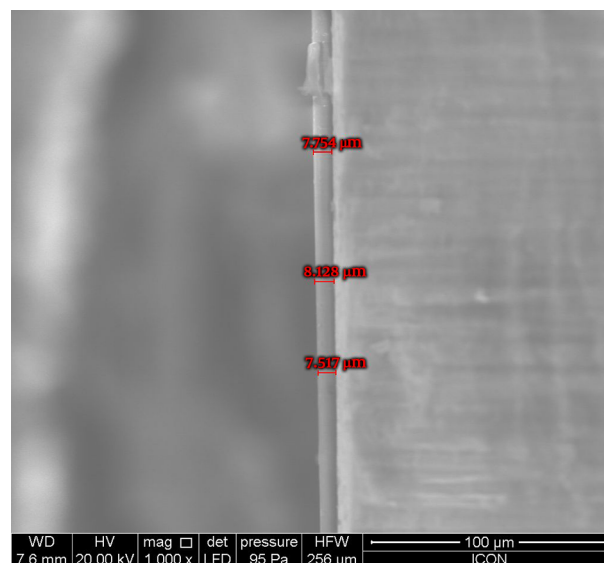


Figure 4. Measurements made of the coating thickness of coated NiTi wires under 1000X magnification

mean values were calculated. Once the mean values for each group were obtained, pre and post exposure thickness was compared for each group (Figure 7).

Pre-exposure thickness for NiTi wires was found to be at 10.636 ± 1.953 microns which was reduced to 8.857 ± 1.599 microns after exposure to artificial saliva. Beta titanium

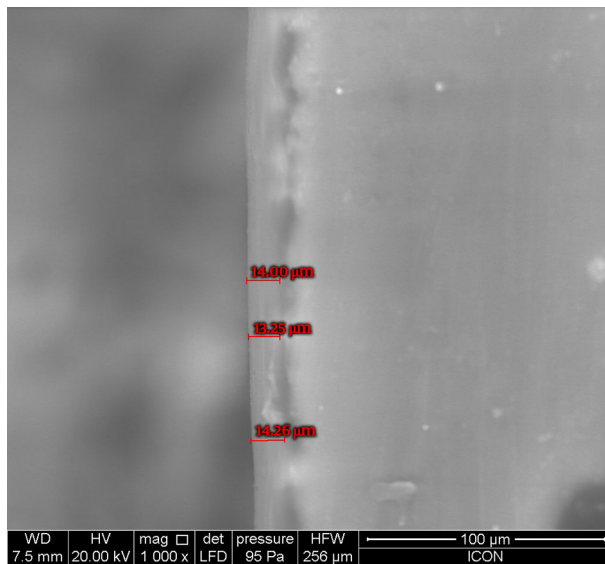


Figure 5. Measurements made of the coating thickness of coated beta-Ti wires under 1000X magnification

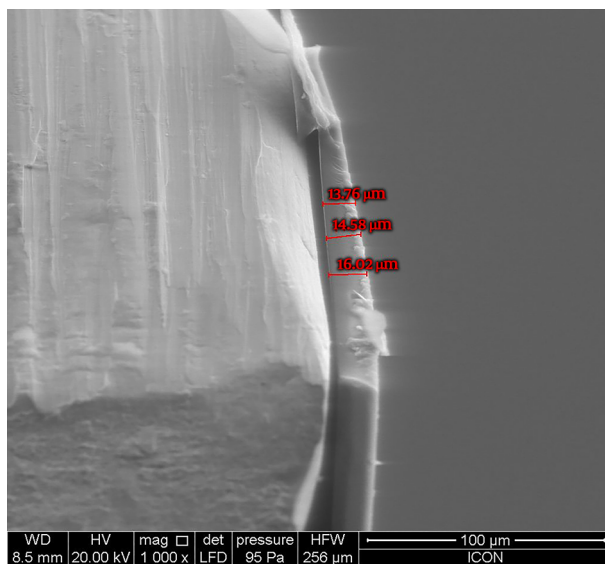


Figure 6. Measurements made of the coating thickness of coated Stainless steel wires under 1000X magnification

wire thickness of 13.498 ± 2.575 microns was reduced to 10.575 ± 2.188 microns and for stainless steel wires, thickness of 13.484 ± 3.223 microns was reduced to 10.638 ± 3.085 microns after exposure to saliva for 21 days.

Thickness was reduced in each group after exposure to artificial saliva which was statistically significant.

4.2. Evaluation of Surface Characteristics

SEM images ($200\times$ magnification) of the nanoparticles coated and uncoated wires before and 21 days after exposure to artificial saliva were obtained.

All six groups showed notable changes in surface morphology. Many of the specimens were characterized by small delamination and irregularities of the coating over some points. The SEM study revealed that the orthodontic wires coated with nanoparticles showed less surface irregularities than the commercially available uncoated wires. Thus the surface topography of the coated wires was found to be smoother with less surface deteriorations as compared to the uncoated wires. The coating layer peeled off leaving surface defects in many areas since the wires were kept immersed in artificial saliva for 21 days while the uncoated wires showed greater degree of surface roughness especially the beta titanium wires group.

4.3. Evaluation of Artificial Saliva for Leaching of Ions

The highest concentrations of ions leached among all the groups were of Iron, followed by silica (Figure 8).

Coated NiTi wires showed lesser concentrations of chromium, iron, nickel and silica when compared with conventional uncoated NiTi wires but the difference was not statistically significant. Coated beta titanium wires showed slightly more concentrations of chromium, iron and nickel when compared with uncoated beta titanium wires while slightly more concentrations of Iron and Silica release were seen from coated stainless steel wires when compared with uncoated stainless steel wires. The net concentration of nickel and the release of nickel were found to be less than chromium.

5. Discussion

Characterization of archwire alloys forms an initial step toward understanding archwire behavior in clinical situations. This helps the clinician to select an appropriate archwire on the basis of the biomechanical requirement of the clinical situation from the plethora of materials available.

The surface topography of an orthodontic wire is an essential property known to influence its mechanical characteristics, esthetic appearance, corrosion behavior, and/or its biocompatibility (9). Various coating techniques and materials have been used with the objective of improving surface properties. However, some difficulty has been faced in this regard, mainly the delamination or wear of the coating (10). The nanoparticle used in our study is Nano Clear which is a commercially available nanoparticle coating precursor solution. It was the solution of choice in our

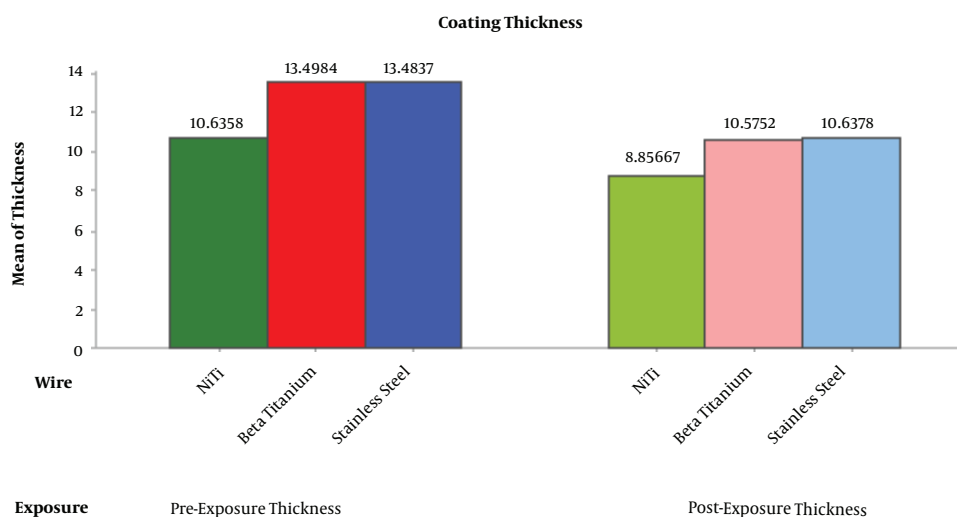


Figure 7. Intergroup comparison for coating thickness of wires

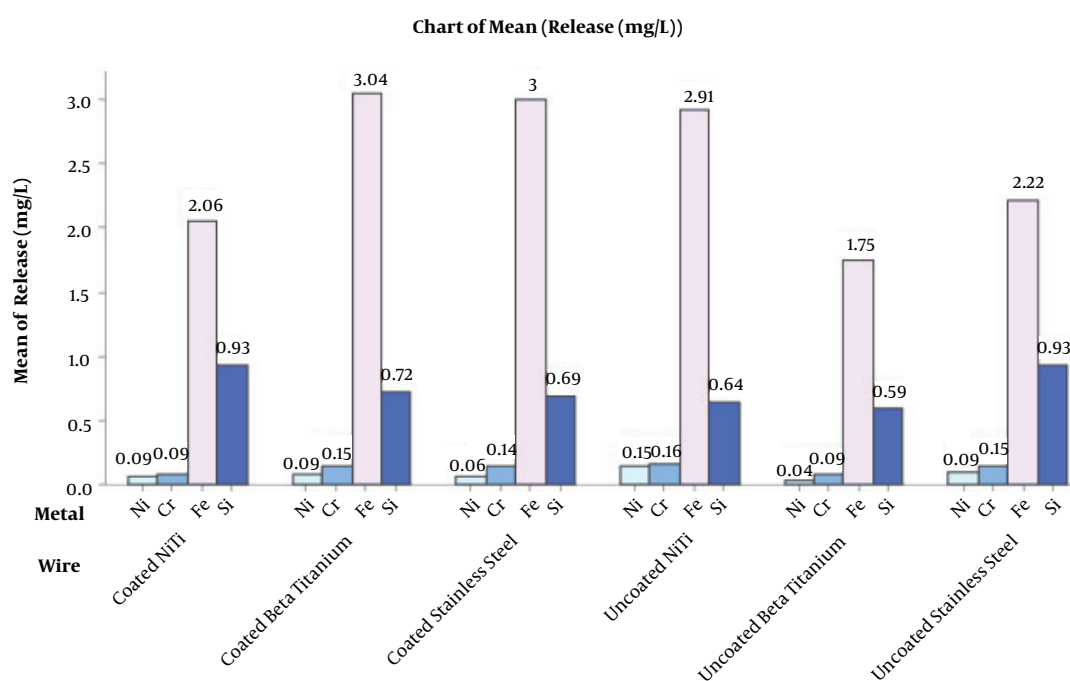


Figure 8. Various ions released from individual groups

study as it is readily available commercially. Secondly, the coating methodology used in our study is a simple procedure which can be performed at the clinical chairside and the coating produces a colorless film which does not alter the appearance of the wire as an aesthetic coating which may produce unaesthetic appearance over time.

The present study is an extension of the research by da

Silva et al. (11) where they evaluated the coating thickness of four brands of as-received esthetic coated rectangular archwires and their surface characteristics and coating stability after 21 days of oral exposure and compared those with conventional stainless steel (SS) and nickel titanium (NiTi) wires. They found that the coated archwires had a low esthetic value as they presented a nondurable coating.

The remaining coating showed severe deterioration and greater surface roughness than postclinical control counterparts. This seems to be in conjunction with the present study where delamination of coating left surface defects and irregularities in many areas since the wires were kept immersed in artificial saliva for 21 days. A great variation in the type and number of surface defects were observed in each sample of coated wires, and also between different samples of uncoated wires, was noted. Additionally, the coating layer clearly tended to be thicker in the center and thinner on the edges which could make the coating layer more susceptible to damage. Besides, this irregularity in coating thickness might impair the wire bracket slot engagement, influencing tooth movement.

Elayyan et al. (10) found similar results where they evaluated *Ex vivo* surface and mechanical properties of coated archwires. In *in vivo* studies conducted by Rongo et al. (12) found similar results where they found SEM images of NiTi wires showing homogeneity for the as-received control wires and a heterogeneous surface with craters and bumps in clinically used esthetic wires. Clinical approaches have also been tried. Demling et al. (13) coated stainless steel brackets with polytetrafluoroethylene (PTFE) and placed them in the oral cavities of children for eight weeks to compare biofilm formation on those brackets vs. uncoated brackets. After this time, a significant reduction in biofilm formation was found in coated brackets.

Nonetheless, investigations continue to find suitable materials and techniques to improve the properties of metallic biomaterials. The increase in friction due to surface roughness is a controversial topic discussed in relevant literature (14-16). Some authors confirm the existence of a close correlation between surface roughness and friction (14) but other studies state that a wire's low surface roughness is not a sufficient condition for low frictional coefficients (10, 16). Nanoparticles have been emphasized to decrease the frictional forces between two metallic surfaces as excellent solid lubricants (17-19). Redlich et al. (18) coated 0.019×0.025 stainless steel orthodontic wires with inorganic fullerene-like nanoparticles of tungsten disulfide (WS₂) and showed significant reduced frictional forces on the wires. Furthermore, the stainless steel orthodontic wires when coated with the nanoparticles of CN_x as suggested by Wei et al. (20) showed significant decrease in frictional forces.

To improve long-term biocompatibility and frictional characteristics, various surface modification methods have been applied to orthodontic appliances (21-24). Diamond-like carbon (DLC) films are promising contributions for this bio-application, owing to their low friction coefficient, chemical inertness and high corrosion resistance (25).

In recent studies, DLC films are widely expected to be adapted as a new biocompatible coating to reduce nickel release from NiTi alloy archwires (22). Kobayashi et al. (22) deposited a diamond-like carbon coating on NiTi archwires to test *in vitro* whether nickel release could be reduced. This investigation concluded that there was a reduction in the concentration of nickel ions in physiological saline, which makes DLC non-cytotoxic in a corrosive environment which seems to be consistent with our findings. Li et al. (26) found similar results where they investigated the biocompatibility of diamond-like carbon (DLC) coated nickel-titanium shape memory alloy (NiTi SMA) *in vitro* and *in vivo* and concluded that diamond-like carbon appears to have better biocompatibility *in vitro* and *in vivo* compared to the uncoated ones. In our study the biocompatibility was tested by evaluating the leaching of ions from both the coated and uncoated wires which was based on the research work by Matos de Souza and Macedo de Menezes (27) where they assessed the *in vivo* release of nickel, chromium, and iron ions into saliva by different metallic brackets and found an increase in the levels of chromium and nickel ions in the saliva which was consistent with our findings.

More toxicity and sensitivity tests, evaluating the effect of the nanoparticles on various body organs and especially oral tissues are necessary.

Limitations of this study include a short duration of exposure to artificial saliva and few studies in the literature to compare our results with, making our conclusions based mainly on our findings. Another limitation was in the evaluation of the coating thickness. The wires were kept stationary in artificial saliva, the role of wire movement and contact with bracket was not considered, which are factors that may affect the stability of the coating. Therefore, the necessity of further knowledge of the surface characteristics of coated archwires is evident.

5.1. Conclusions

The present study has identified important changes in the properties of coated archwires after exposure to artificial saliva in an attempt to simulate oral environment. The exposure to artificial saliva for 21 days had an important role in the coating loss and in the surface quality of coated wires. It provides critical information concerning the performance of materials in the environment in which they are intended to operate. In addition, this method helps in the assessment of alterations of material properties that can occur during clinical use. With further improvements in coating method and its approval for use in the oral cavity, the problem of friction during orthodontic treatment could be minimized consequently, enhancing anchorage

control and direction of tooth movement, reducing duration of the treatment and decreasing the risk of root resorption.

Footnotes

Conflict of Interests: It is not declared by the author.

Financial Disclosure: Authors have no relevant financial interests related to the material in the manuscript.

Funding/Support: It is not declared by the author.

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