

Surface Topography and Composition of As-received and-retrieved Initial Archwires: A Comparative Study

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ABSTRACT

Aim: To compare the surface topography and composition of As-received and retrieved initial archwires using scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDS).

Materials and methods: The as-received round 0.016-inch stainless steel and nickel titanium archwires were taken from wire packets. The surface and composition of retrieved 0.016-inch stainless steel and nickel-titanium wires ($n = 15$), in service intraorally for at least 2 months, were compared using SEM and EDS.

Results: The SEM images of As-received wires showed surface irregularities. The As-received stainless steel wire was found to be rougher than the As-received nickel titanium wire. In comparison with the As-received wire, the retrieved stainless steel archwires revealed deeper grooves, pits, and areas of corrosion. The retrieved nickel titanium wires, on the comparison, revealed no appreciable difference. The EDS analysis showed leaching of iron, nickel, and chromium in stainless steel wires and leaching of nickel in nickel-titanium wires. Oxygen and carbon concentrations were increased in both.

Conclusion: There does occur a change in the surface topography and composition of wires after use intraorally. Changes were more appreciable in stainless steel wires than nickel-titanium wires. Leaching and deposition of surface elements need to be correlated with the toxic human levels.

Clinical significance: Surface topography and surface roughness of the orthodontic archwires affect the efficacy of orthodontic treatment. This study will try and elicit the qualitative and quantitative changes in the initial archwires with respect to surface topography and surface roughness and also attempt to shed some light on the ways to minimize any alterations.

Keywords: Archwires, Energy dispersive X-ray spectroscopy, Nickel-titanium, Scanning electron microscope, Stainless steel, Surface topography. World Journal of Dentistry, (2019): 10.5005/jp-journals-10015-1621

INTRODUCTION

Tooth movement in orthodontics is brought about by orthodontic archwires, which generate the biomechanical forces, transmit these forces via brackets to bring about tooth movement. Stainless steel (SS) and nickel-titanium (NiTi) wires are the most commonly used orthodontic archwires these days.¹

The surface topography of orthodontic wires is an important trait known to affect the mechanical property, the biocompatibility and corrosion.² The structure of the surface of the wire is dependent on the type of alloy used, manufacturing process and the surface finish treatment.³ Orthodontic wires are constantly engaged in the brackets using ligatures or modules and therefore make favorable sites for corrosion. Corrosion resistance is of utmost importance for orthodontic wires as corrosion can cause surface roughening, appliance weakening, and release of elements from the alloy. Orthodontic wires containing nickel have been implicated to cause a type IV delayed hypersensitivity immune response, due to the leaching of nickel ions intraorally. It also increases the friction between the archwire-bracket interface due to increased surface roughness.

The method for the analysis of the surface morphology of orthodontic materials commonly used is a scanning electron microscope with energy-dispersive X-ray spectroscopy. The SEM-EDX analysis enables both qualitative and quantitative assessment of constituent elements in the alloys along with an indirect estimation of the ion release from the nickel, chromium, and iron of the orthodontic alloys.⁴

The aim of the current study was to compare the surface topography and composition of As-received and-retrieved initial archwires using scanning electron microscope and energy dispersive X-ray spectroscopy.

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MATERIALS AND METHODS

The As-received wire group comprised of an unused wire each of Prime Ortho NiTi and Prime Ortho stainless steel having 0.016-inch cross-section. The retrieved nickel-titanium and stainless steel wires included in the study were in service for a minimum of 2 months in the oral cavity of 30 patients (a group of 15 for each wire type) and were collected during the routine orthodontic visits of the patients to the Department of Orthodontics and Dentofacial Orthopedics at Maharishi Markandeshwar College of Dental Sciences and Research.

All retrieved wires had a cross section of 0.016-inch and had been ligated to 0.022 slot metal brackets using elastomeric modules or stainless steel ligatures. This study was approved by the Committee of Ethics in Research.

Cleaning Procedure

After retrieval, the wires were cleaned in ultrasonic cleaner similar to the cleaning procedure followed by Daems et al.³ The As-received and retrieved stainless steel, and NiTi wires were first ultrasonically cleaned with 1N sodium hydroxide (60°C for 15 minutes) to remove organic matter, and then rinsed using distilled water. Next, the samples were cleaned ultrasonically with 4% sulphuric acid (60°C for 15 minutes) to remove inorganic debris and rinsed using distilled water. Similar steps were followed for the As-received wires.

Experimental Procedure

The specimens (10 mm) for examination were taken from the anterior region of the wires and mounted on aluminum stubs. The surface of as-received and retrieved stainless steel and NiTi wires were examined using Scanning Electron Microscopy [Carl Zeiss EVO40 (Cambridge, UK) used at 20 Kv] with Energy Dispersive X-ray Spectroscopy [Bruker X-Flash detector 4010 (Germany)] at Advanced Institute of Research Facility, Jawaharlal Nehru University, New Delhi. Surface characteristics were evaluated according to the visual assessment of the surface defects.

RESULTS

Scanning Electron Microscopic Findings

As-received Stainless Steel Wires (Fig. 1)

The As-received stainless steel wires displayed a surface which was smooth with lines parallel to the long axis of the wire which

can be attributed to the drawing procedure done during the manufacturing process of the wires. Minor scratches and pits were also present suggesting chemical interactions during manufacturing.

Retrieved Stainless Steel Wires (Fig. 2)

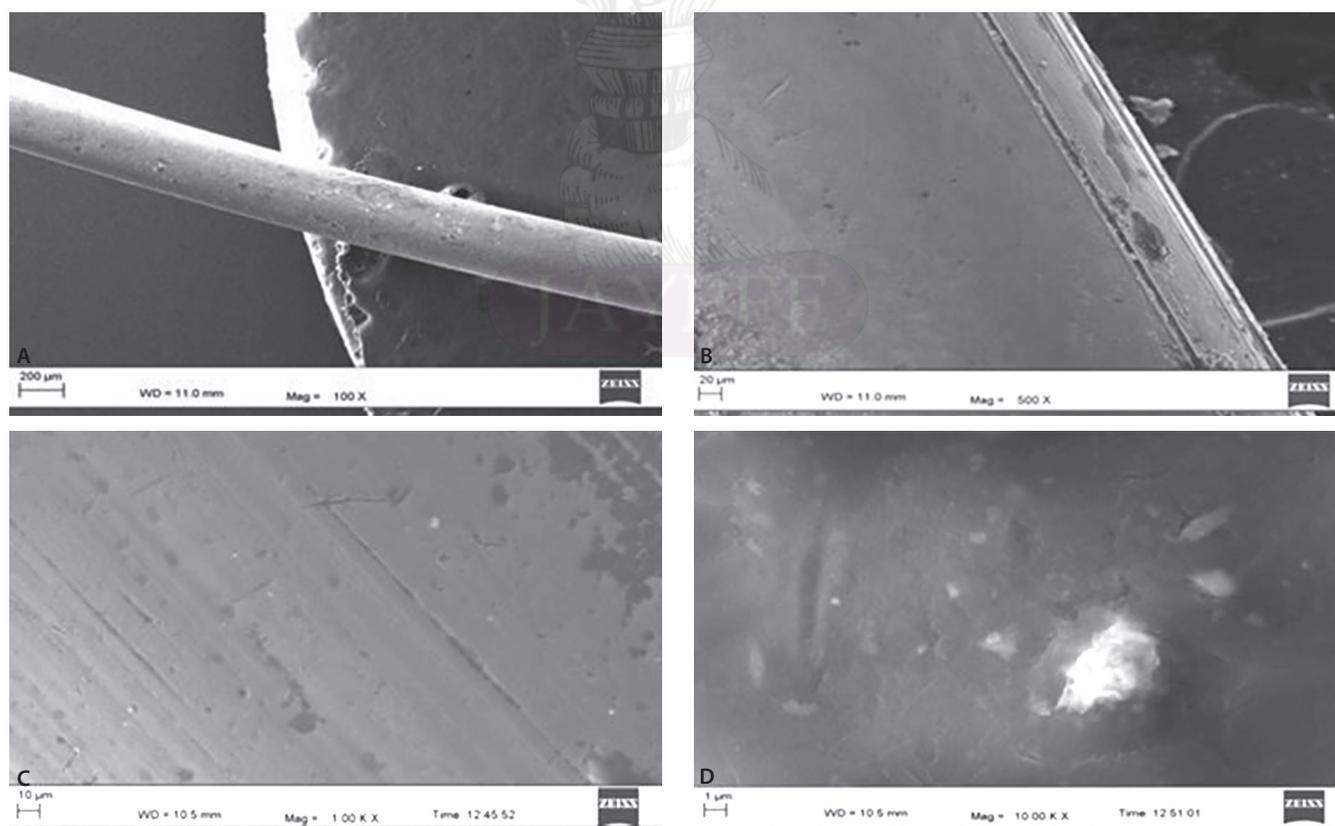
The retrieved stainless steel wires displayed striations and grooves along the longitudinal axis of the wires which appeared to be deeper than the As-received wires. There were also small pits and scratches running perpendicular to the drawing direction of the wire. Numerous dark patches depicting areas of corrosion were seen.

As-received Nickel-Titanium Wires (Fig. 3)

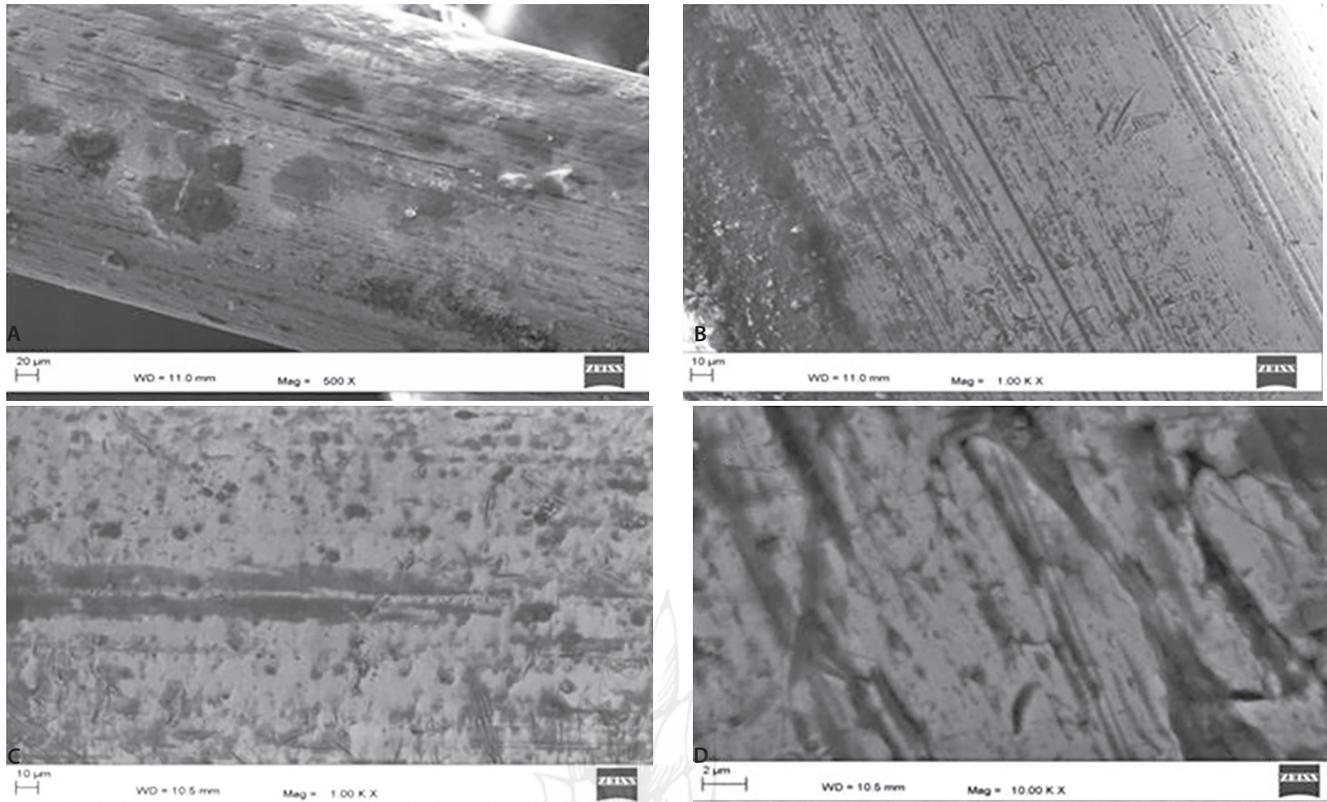
The As-received NiTi wires also displayed a smooth surface with lines parallel to the longitudinal axis of the archwire which can be attributed to the drawing procedure during the manufacturing process of the wires. Minor scratches and pits were also present suggesting chemical interactions during manufacturing.

Retrieved Nickel Titanium wires (Fig. 4)

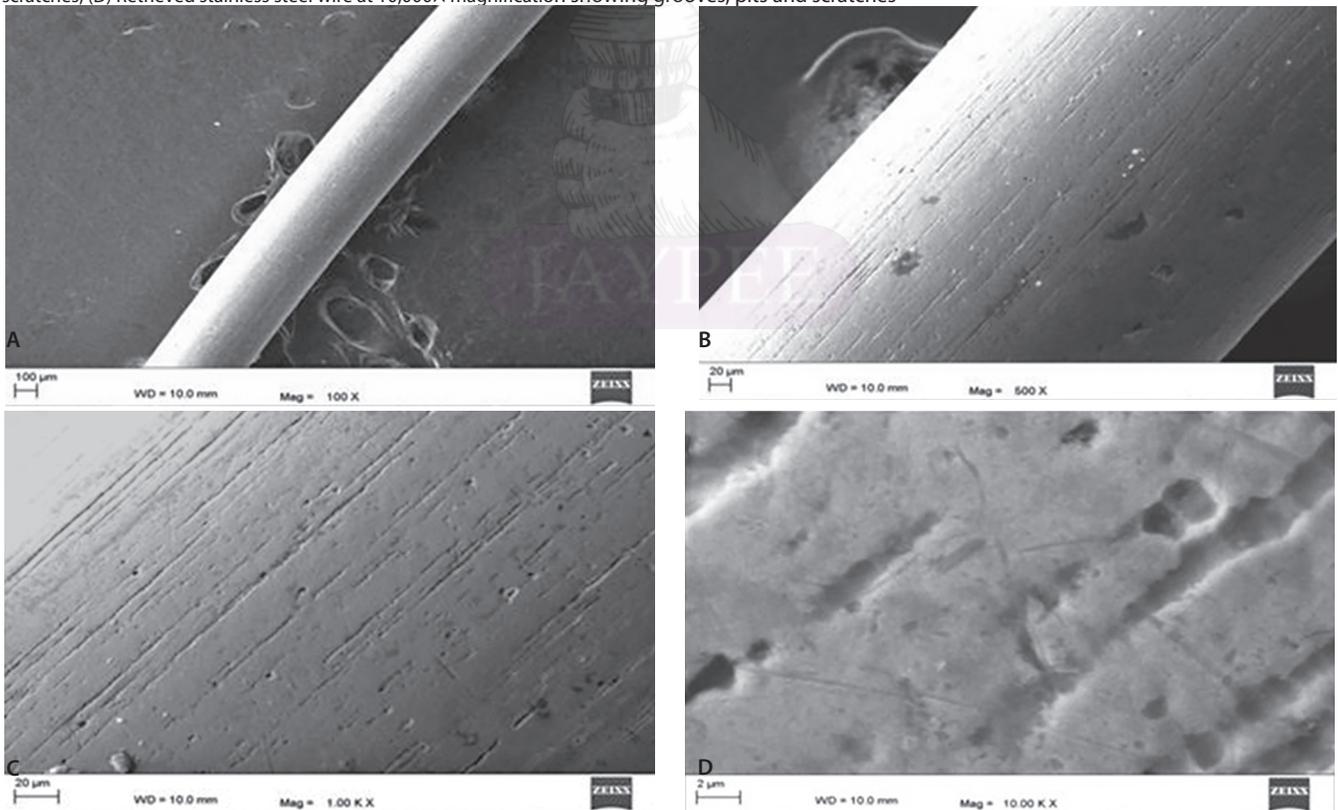
The retrieved nickel titanium wires were smooth but displayed striations and grooves along the longitudinal axis of the wires which appeared to be similar to the As-received wires. There were also small pits and scratches spread throughout the wire. Few samples showed dark patches depicting areas of corrosion. Overall, the difference between the As-received and retrieved NiTi wire surfaces was not greatly appreciable.



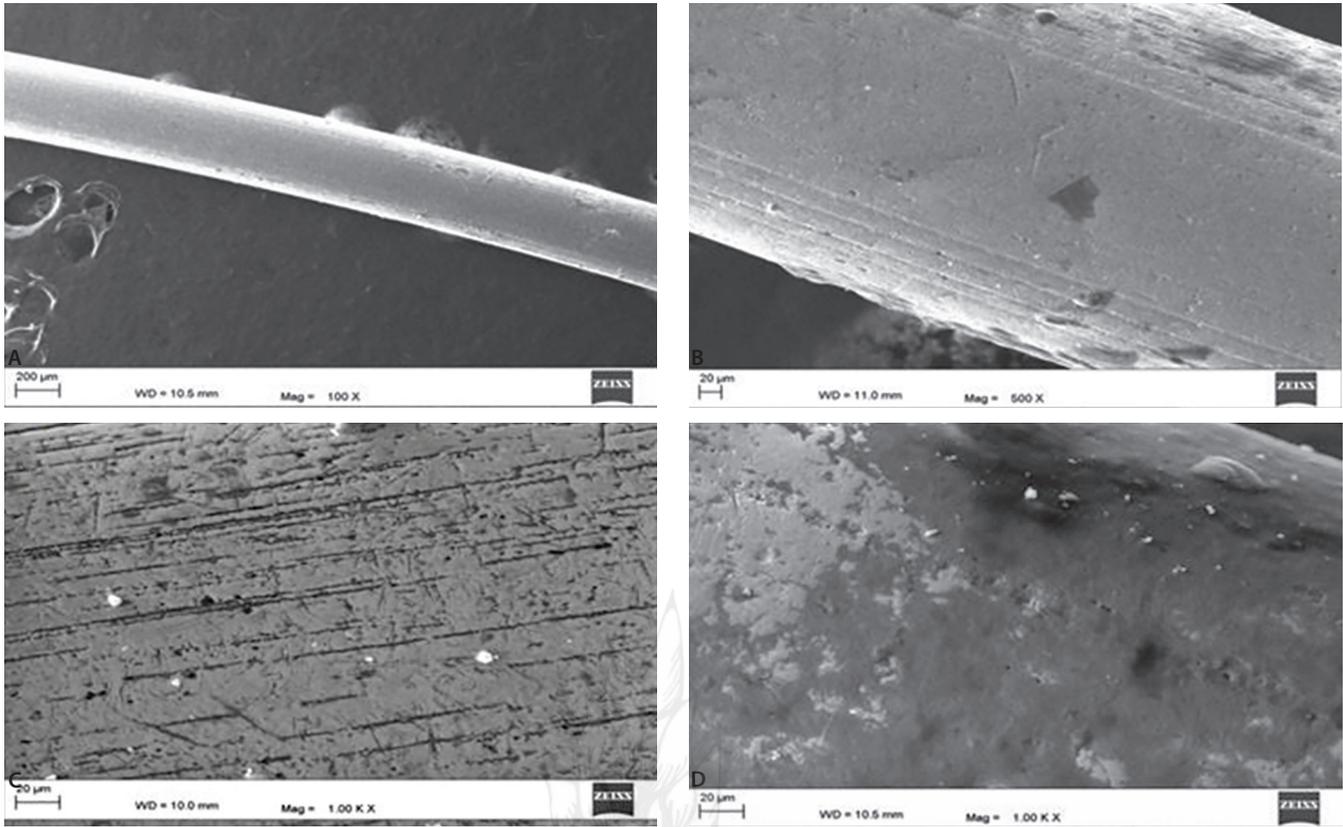
Figs 1A to D: (A) As-received stainless steel wire at 100× magnification showing a smooth surface; (B) As-received stainless steel wire at 500× magnification showing a smooth surface with minor scratches and pits; (C) As-received stainless steel wire at 1000× magnification showing lines parallel to long axis of the wire; (D) As-received stainless steel wire at 10,000× magnification showing minor scratches and pits



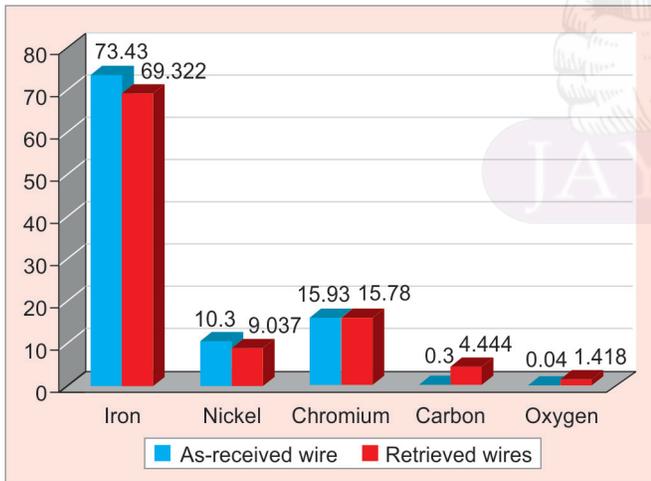
Figs 2A to D: (A) Retrieved stainless steel wire at 500X magnification showing longitudinal grooves, pits and scratches and areas of corrosion; (B) Retrieved stainless steel wire at 1000X magnification showing striations and grooves along the long axis; (C) Retrieved stainless steel wire at 1000X magnification showing pits and scratches; (D) Retrieved stainless steel wire at 10,000X magnification showing grooves, pits and scratches



Figs 3A to D: (A) As-received nickel titanium wire at 100X magnification showing a smooth surface; (B) As-received nickel titanium wire at 500X magnification showing a smooth surface with minor scratches and pits with lines parallel to long axis of the wire; (C) As-received nickel titanium wire at 1000X magnification showing lines parallel to long axis of the wire along with pits and scratches; (D) As-received nickel titanium wire at 10,000x showing minor pits, grooves and scratches.



Figs 4A to D: A) Retrieved nickel titanium wire at 100× magnification showing a smooth surface; B) Retrieved nickel titanium wire at 500× magnification showing pits and scratches; C) Retrieved nickel titanium wire at 1000× magnification showing pits and scratches and lines parallel to long axis; D) Retrieved nickel titanium wire at 10,000× magnification showing lines parallel to long axis alongwith grooves alongwith pits and scratches

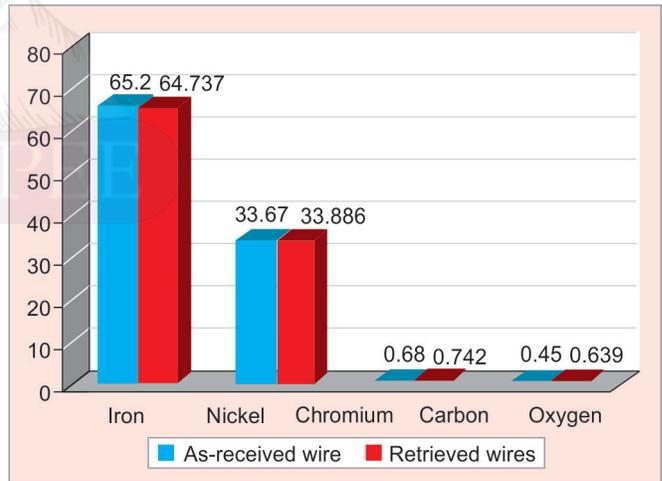


Graph 1: Comparison of changes in elemental composition of As-received and retrieved stainless steel arch wires using EDS

Energy Dispersive X-ray Spectroscopy Observations

A comparison between the composition of As-received and retrieved stainless steel wires revealed a decrease in the mean concentration (wt%) of iron, nickel and chromium and an increase in carbon and oxygen concentration (wt%) (Graph 1).

A comparison between the composition of as-received and retrieved NiTi wires revealed a decrease in the mean concentration



Graph 2: Comparison of changes in elemental composition of As-received and retrieved nickel titanium arch wires using EDS

(wt%) of nickel and an increase in titanium, carbon and oxygen concentration (wt%) (Graph 2).

Statistical Analysis

Student’s t-test was applied, and the differences were found to be statistically significant ($p < 0.05$) for all the elements except titanium whose mean concentration although was found to be increased but the increase was not statistically significant.

DISCUSSION

Retrieval analyses studies (study of material after use intraorally) have gained importance in biomaterials research since vital information is obtained from assessing the behavior of a product in the environment where it actually functions.⁵ The retrieved NiTi and stainless steel (SS) wires used in our study were functioning in the oral cavity of patients for at least 2 months. After retrieval, the wires were cleaned in ultrasonic cleaner similar to the cleaning procedure followed by Daems et al.³

The As-received and retrieved wire specimens (10 mm) were then mounted on aluminum stubs and viewed in a scanning electron microscope at 100x, 500x, 1000x and 10000x magnifications.

The results demonstrated that the As-received stainless steel wires displayed a smooth surface with lines running in line with the long axis of the wire. This observation was in agreement with other studies^{1,3,6} done to assess the unused wire surface. This topographical feature can be attributed to the drawing procedure during the manufacturing process of the wires. Minor scratches and pits were also present which could be due to chemical interactions during manufacturing as suggested in the study by Daems et al.³ who evaluated the material degradation of unused and used stainless steel archwires using SEM.

The retrieved stainless steel wires, on the other hand, displayed deep striations and grooves along the longitudinal axis of the wires and also small scratches running perpendicular to the drawing direction of the wire. Numerous dark patches depicting areas of corrosion were seen. The findings were in concordance with other studies^{5,7,8} wherein the changes in wire surface were examined post intraoral use^{3,5} after immersion in an electrolyte solution of varying pH^{7,9} or artificial saliva.⁸

It was concluded that surface irregularities occur due to orthodontic handling, the effect of pH and interaction with plaque and food ultimately leading to pitting corrosion. Thus, on a comparison of the SEM images of as-received and retrieved stainless steel (SS) archwires, a clear difference in their surface topographies was appreciated. The findings are in agreement with studies conducted by Daems et al.³ and Kararia et al.⁵ who found that the surface defects formed during the process of manufacturing got altered or intensified post intraoral use. But Edie et al.,¹⁰ Grimsdottir,¹¹ and Premanand et al.¹² found no or very few changes after use intraorally^{10,11} or after simulated tooth movements *in vitro*.¹²

The As-received nickel titanium wires also displayed a smooth surface with lines parallel to the longitudinal axis of the archwire which can be attributed to the drawing procedure during the manufacturing process of the wires. This was in concordance with other studies^{1,11,13,14} which assessed the surface of As-received wires from different manufacturers. Minor scratches and pits were also present suggesting chemical interactions during manufacturing.^{11,13,15}

In comparison, the As-received NiTi wire surface appeared to be rougher than the As-received SS wire surface. A similar observation was made by Amini et al.¹ and Yu et al.² in which they concluded that unused NiTi wire was more irregular than unused SS wire. It was suggested that the surface properties of the NiTi wires having deeper pits might be the consequence of their complicated manufacturing processes.¹

The retrieved NiTi wires were smooth but displayed striations and grooves along the longitudinal axis of the wires which appeared to be similar to the As-received wires. There were also small pits and scratches spread throughout the wire. Few samples showed dark

patches depicting areas of corrosion. The results were in agreement with other *ex vivo*¹⁶⁻¹⁸ and *in vitro*¹⁹⁻²¹ studies on used wires. Overall, the difference between the as-received and retrieved NiTi wire surfaces was not greatly appreciable. No discernible difference was found in the studies by Edie et al.¹⁰ and Grimsdottir¹¹ on a comparison of NiTi wires before and after clinical service.

Research on the intraoral alterations of orthodontic archwires has revealed a wide array of degradation phenomena.¹⁶ The effect of the oral environment as well as of the resulting corrosion on the chemical composition of As-received and retrieved orthodontic wires was assessed in our study using energy dispersive X-ray spectroscopy (EDS). EDS determines the elemental constitution of material on interaction with X-rays, depending on the energy differences that occur during excitation and downfall of its electrons.¹³

The elemental composition (wt%) of As-received wire was compared with the mean elemental composition (wt%) of the retrieved wire samples. The comparison of SS wires revealed a decrease in the mean concentration (wt%) of iron (Fe), nickel (Ni) and chromium (Cr) and an increase in carbon (C) and oxygen (O) concentration (wt%). All differences were statistically significant.

The comparison of NiTi wires revealed a decrease in the mean concentration (wt%) of nickel (Ni) and an increase in titanium (Ti), carbon (C) and oxygen (O) concentration (wt%). The differences were statistically significant for all the elements except Titanium whose mean concentration (wt%) although was found to be increased, but the increase was not statistically significant.

The increased levels of oxygen on both used SS and used NiTi wires were consistent with a study by Edie et al.¹⁰ They suggested that an adherent oxide layer forms on the wires as a consequence of subjection to an aqueous environment, and the formation of this oxide layer depends to some extent on surface characteristics.¹⁰ An increase in oxygen levels was also seen by Santos et al.²² who studied the effect of time and pH on stainless steel wires and brackets and also by Mikulewicz⁴ who suggested it to be due to passivation by oxides.

An elevated level of carbon on both used SS and used NiTi wires was seen. Santos et al.²² also found increased carbon content on stainless steel archwires, and the increase was in proportion to intraoral time. Toker and Canadinc²³ evaluated the biocompatibility of NiTi wires using EDS and found that the retrieved wires showed the presence of carbon-containing compounds. They suggested that the development of huge deposits of these structures containing carbon is associated with physical aspects instead of the chemical constitution of saliva.

There was a significant decrease in the iron content of retrieved stainless steel wires in this study. This decrease was also seen in studies by Mikulewicz⁴ who suggested solubilization of iron due to corrosion and Santos et al.²² who concluded that the presence of iron was indirectly proportional to time. Toker,²³ and Gopikrishnan et al.²⁴ conducted *in vitro* studies using artificial saliva, and they also observed a decrease in iron concentration of the wires, but in both the studies the iron release was below toxic levels.

In our study, retrieved stainless steel wires show a decreased mean concentration (wt%) of chromium. Mikulewicz et al.⁴ in a similar study suggested solubilization of chromium as the cause of decreased concentration. Santos et al.²² concluded that the presence of chromium was indirectly proportional to time. Complexometric titration of used wires done in a study by Kararia et al.⁵ also revealed significant release of chromium ions. *In vitro* studies by Toker,²³ and Gopikrishnan et al.²⁴ also showed chromium ion release from wires.

In our study, there was a significant decrease in the mean concentration (wt%) of nickel in both-retrieved stainless steel and retrieved Nickel Titanium wires. These results were in concordance with a number of other *ex vivo*^{20,22,25} and *in vitro*^{23,24,26} studies in which leaching of nickel has been demonstrated from used SS and NiTi archwires. However, no variations were found with respect to Ni concentration ratios on a comparison of As-received and retrieved NiTi or SS wires in a study done by Eliades,²⁷ although they stated that the results from this study should not be regarded as a definitive indication of the lack of ionic release from alloys.

The previous retrieval studies indicate the concentration of nickel and chromium as ratios to iron and titanium in stainless steel and NiTi wires, respectively. The present study gives the concentration of the all the elements and not as a ratio as the comparison must be drawn between As-received and retrieved wires to observe changes brought about by the oral environment on all the constituent elements.

This study shows that there does occur a change in overall elemental composition of retrieved NiTi and SS archwires when they were compared with As-received wires with leaching of chromium, iron, and nickel from the stainless steel wires and chromium and nickel from the NiTi wires. These findings must be correlated with the toxic levels in the human body.

The current system for regulation of general instruments, materials, and equipments is based on the International Standard Organization (ISO) The ISO/CD 15841:2014 is a revised version of the international standard for archwires. But their implementation is not compulsory for vendors or manufacturers. All the manufacturers are allowed to devise their specifications for their products. Hence, one lot of wire can vary from another from the same manufacturer.⁶

Taking this lack of standardization into consideration, one limitation of this study was that there was only one sample used in the as-received group to assess the elemental composition.

Further studies need to be done to assess the correlation of topographical changes with severity of crowding, food habits of the patients, duration of intraoral aging, methods of ligation and other factors and to correlate the amount of leaching with the toxic levels of these elements in the human body.

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