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Characteristics of Sterilised Nickel Titanium and Stainless Steel Orthodontic Archwires

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ABSTRACT: *Sterilisation removes microorganisms in the vegetative or spore states from a material, surface or medium. Ensuring infection control is of utmost importance for health safety. Pathogenic microorganisms are frequently transmitted from one person to another because of ineffective sterilising agents, apparatus and equipment. The aim of this study is to investigate the effect of sterilisation methods on morphological parameters of super elastic nickel titanium (NiTi) and stainless steel (SS) orthodontic archwires. The archwires were sterilised using a variety of methods, including dry heat sterilisation (oven) at 160°C for 1 h, steam heat sterilisation (autoclave) at 121°C for 15 min and cold sterilisation (2% glutaraldehyde) at 10 h. After sterilisation, the wires were tested, and the morphological changes was determined through atomic force microscopy (AFM), scanning electron microscopy (SEM) and X-ray diffraction (XRD). The AFM and SEM results obtained before and after the sterilisation of the NiTi and SS orthodontic archwires did not show clear differences in the surface structure. No changes in the elemental composition of the NiTi and SS wires, and no new phase appeared according to the XRD* *results which support the use of archwire sterilisation procedures as part of the infection control treatment and are considered a suitable procedure related to the characterisation of the orthodontic archwires.*

Keywords: sterilisation method, super elastic NiTi, stainless steel wire, archwire, surface morphology

1. INTRODUCTION

Sterilisation is an essential step in the production of any biomaterial or medical equipment that will come into contact with the human body and in the reuse of medical instruments.[1] The American Dental Association Council on Dental Materials (1986) recommends using an autoclave, chemical vapour or dry heat to sterilise metal and heat-stable instruments. According to the Centres for Disease Control, all tools that come into contact with the oral mucosa must be sterile. Dry-heat sterilisation, also known as hot-air-oven sterilisation, utilises temperature of 160°C and holding period of at least 2 h.[2] At temperatures higher than 190°C, the holding time is set at 6 min for unwrapped objects. As for wrapped objects, a 12 min holding time is recommended. Cold sterilisation involves the use of chemicals and solvents as disinfectant agents, such as glutaraldehyde, formaldehyde, ortho-phthalaldehyde, peracetic acid and hydrogen peroxide at concentrations ranging from 0.2% to 10%.[2] Steam sterilisation is mostly employed with an autoclave, and the recommended temperature and holding time ranges from 15 min at 121°C to 3 min at 134°C, respectively.[2]

Orthodontic archwires are commonly marketed, and cross-contamination is prevented by wrapping archwires individually in separate sealed packets with or without pre-sterilisation.[3] Archwire manufacturers recommend sterilising the wires to prevent cross-contamination that may lead to the production of undesirable biomechanical forces that can affect progressive tooth movement during treatment.[4] All sterilisation methods effectively eliminate contamination by bacteria from orthodontic appliances, ensuring patient's safety.[3] However, the effects of changes in the physicochemical and mechanical properties of archwires after sterilisation have presented considerable concern. Surface roughness is an important measure of mechanical product quality because it accurately represents various characteristics, such as fatigue strength, wear resistance and surface hardness related uniquely to orthodontic archwires. It refers to the spacing between microscopic peaks and valleys on the surface of a workpiece, reflecting the microscale unevenness of the surface.[5]

Orthodontic archwires fabricated from various alloys, such as stainless steel (SS), nickel titanium (NiTi), copper NiTi and β-titanium (β-Ti), are suitable alternatives for wire changes during treatments.[6] SS archwires are highly utilised during orthodontic treatment, although NiTi and β-Ti are progressively attracting interest because NiTi wires possess super elastic (SE) and shape-memory properties.[7]

The surface structure of archwires is determined by alloys incorporated during manufacturing and resulting surface finishing.[4] An orthodontic archwire's surface topography is an important feature that influences its mechanical properties, aesthetic results, corrosion behaviour and biocompatibility with appliances.[8] However, conflicting results about the effects of sterilisation on the surfaces and characteristics of NiTi archwires commonly utilised during orthodontic treatment have been reported.[4,9] Steam and dry-heat sterilisation lead to a low load–deflection properties for the loading and unloading forces of NiTi archwires, thus reducing forces applied to archwires.[9] NiTi archwires have large numbers of irregularities on their surfaces before sterilisation and clinical use but become smooth upon clinical recycling and repeated sterilisation.[10] Presence of friction between an archwire and bracket during sliding mechanics increases with surface roughness and surface irregularities render the archwires prone to corrosion and may lead to plaque accumulation.[10] Surface roughness can affect the frictional coefficient between an archwire and bracket slot, leading to interference while the archwire slides through each tube and bracket during orthodontic treatment, obstructing the bracket from sliding along the wire correctly and delaying or preventing tooth movement.[11] Furthermore, the surface roughness of orthodontic archwires can reduce aesthetic features and corrosion resistance.[12] Sterilisation and disinfection can cause changes in physical and mechanical properties.[9,13] However, whether these effects are beneficial or detrimental to clinical applications is unclear.[14] Hence, evaluating the performance of orthodontic archwires on the basis of their surface roughness especially after sterilisation is crucial to the success of orthodontic treatment.[12] Initially, surface profilometry is considered the primary method for assessing the contours, shapes, grooves and surface roughness of orthodontic archwires.[12] Currently, many methods for assessing surface roughness have been used, such as scanning electron microscopy (SEM) with energy-dispersive spectroscopy (EDS), X-ray diffraction (XRD), differential scanning dilatometry, atomic force microscopy (AFM) and digital stereomicroscopy.[6,15,16] AFM is suitable for investigating surface topography because it is flexible in defining measured areas.[17] Thus, we adopted different methods to analyse surface roughness and assessed the effects of different sterilisation methods, namely, dry-heat sterilisation, steam sterilisation with an autoclave and cold sterilisation with 2% acidic glutaraldehyde, on the surface characteristics of SS and SE NiTi archwires. AFM, SEM with EDS and XRD were performed for further analysis.

2. EXPERIMENTAL

2.1 Archwire Preparation

A pre-packed orthodontic archwire has an arch shape with a wide U-shaped at the centre part and straight distal ends. This study used SE NiTi (N = 12) and SS (N = 12) round archwires with diameters of 0.016 inches (International Orthodontic Service [IOS], USA) to determine morphological changes before and after sterilisation. For each group, a total of 24 archwires were obtained by cutting approximately 5 cm along the length of the straight distal portion of both ends of each orthodontic archwire (see Figure 1).[18] The accuracy of each wire dimension was measured with a digital vernier calliper (IOS, USA). The archwires were divided into four groups. One group was the control (N = 3, no treatment), and the other three groups consisted of wires sterilised through dry-heat sterilisation (oven) at 160°C for 1 h (N = 3), steam-heat sterilisation (autoclave) at 121°C for 15 min (N = 3) and cold sterilisation (2% glutaraldehyde) for 10 h (N = 3).

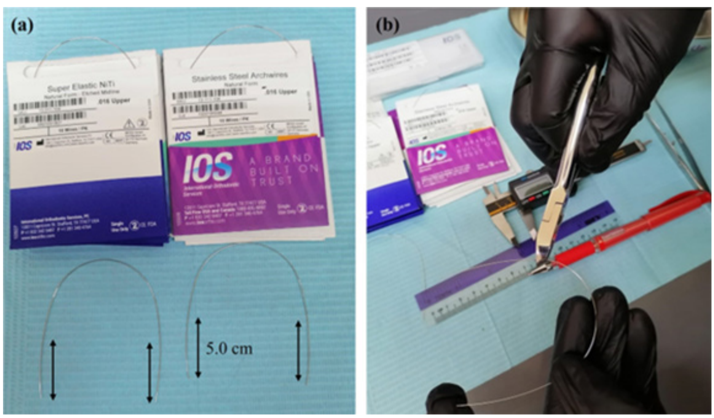

Figure 1: Samples (a) were obtained by cutting both distal ends of each archwire (see [b]).

2.2 Sterilisation Procedure

Sterilisation was carried out in the Dental Laboratory at the College of Health and Medical Techniques, Department of Prosthetics of Dental Techniques (Iraq, Baghdad). The samples were ultrasonically cleaned in distilled water for 20 min and sterilised using three methods: (1) In dry-heat sterilisation, the wires were placed on a sterilisation pan of an oven steriliser (Memmert, West Germany) and left exposed for sterilisation. After preheating, the temperature of the steriliser was maintained at 160°C for 1 h, and the wires were cooled for 15 min at room temperature. (2) In autoclave sterilisation, the wires were individually packaged in clear sterilisation bags. Steam-heat sterilisation was performed at 121°C and 15 PSI for 15 min in an autoclave (Tanda Steam Steriliser, Wosow Medical, China). In an autoclave cycle, contact between different types of wires was prevented by spreading them on an autoclave tray. This procedure also ensured that steam can freely circulate through each wire. At the end of the autoclave cycle, the wires were individually dried and allowed to cool at room temperature. (3) In the cold sterilisation method, wires were placed in freshly prepared 2% acidic glutaraldehyde (China) for 10 h according to the manufacturer's instructions. After cold sterilisation, the wires were rinsed with running water, placed on an absorbent paper and air-dried at room temperature. Then, the wires were packed in an airtight cover, and the surface parameters' behaviour were determined within 1 week after sterilisation.

2.3 Surface Characterisation Analysis

Surface characterisation analysis was performed on the basis of differences in the surface morphology, surface roughness and crystal structures of samples. The three-dimensional (3D) AFM images of all samples' surfaces (Naio AFM 2022 model, Nanosurf AG, Switzerland) were used in determining rough surfaces. Micromorphology and characteristics were determined using a field-emission SEM system (Inspect F 50, Fei Company, Czech Republic), and the distribution map of the elements on a surface was established through EDS and XRD analysis and utilised in investigating crystal structures formed on the surfaces (Bruker, Germany). The data were reported as mean and standard deviation, and dependent variables were analysed using one-way ANOVA and Scheffe test. A *p*-value of < 0.05 indicated statistical significance. For each variable, comparison was made between untreated control NiTi and SS archwires because archwires are expensive and the remaining wide U-shaped archwire cannot be reused for other experimental work.

2.3.1 Atomic force microscopy

The surface roughness of the archwire segments was measured using an AFM system (Naio AFM 2022, Nanosurf AG, Switzerland) located at the General Service Laboratory, Department of Chemistry, College of Sciences, University of Baghdad. The surface roughness of the wires was measured through AFM at a scan area of 10 μm × 10 μm. A small portion (3 mm) of each wire segment was mounted on a double-sided tape. The AFM probe used in contact mode for measurements was diamond-like carbon (C) coated on the tip side of the cantilever and 15 nm-thick aluminium on the detector side. The force constant of the cantilever ranged from 0.07 N/m to 0.4 N/m, and the probe specifications were as follows: beaming shape length, 450 μm; beaming shape width, 50 μm; and resonance frequency, 13 kHz.[19] The samples were scanned at room temperature from three regions: the middle and each end of the wire. The mean value of the three measurements was determined, which represented the root mean square (RMS) value.[20,21] The surface 3D image of the archwire section was shown on the monitor of the attached computer and was processed and analysed using proprietary software supplied with the AFM device.[22]

2.3.2 Scanning electron microscopy

The micromorphological archwires characteristics were evaluated using SEM at different magnifications, and photographs were recorded at ×2,500 magnification (Alkhora Company Lab, Baghdad, Iraq). Each sample was fixed on a sample holder and viewed under an SEM system (Inspect f 50, Fei Company, Czech Republic). The wires were coated with a 10 nm-thick layer of gold with a sputter coater machine (YKY Company, China), and vapour deposition was performed to make the surface electron opaque. To obtain SEM images, we exposed the samples to electrons and analysed the reflected electron intensity on each pixel. The surface properties were evaluated through the visual examination of surface irregularities. The system was equipped with an EDS device for analysing the chemical composition of each wire. The X-ray energy spectrum dispersed from the surface was analysed for the identification of the wire materials' composition.

2.3.3 X-ray diffraction

An XRD system (Bruker, Germany) was used in investigating crystal structures, types of orientations and boundaries among crystals.[23] The archwires were mounted on a glass slide with clay, and diffraction measurements were obtained using a standard coupled scan. The scan began at a 10° (2θ) and ended at a 90° angle at a step size of 1° and count time of 6 s, and XRD data were plotted using Siemens software. The obtained phases were identified by comparing the XRD peaks with the d-spacing in JCPDS files (18-899; Joint Commission on Powder Diffraction Standards, International Centre for Diffraction Data, Swarthmore, Pa).

3. RESULTS AND DISCUSSION

3.1 Surface Roughness of Sterilised Orthodontic Archwires

Through AFM measurements, the wires were categorised according to their average roughness (Ra), RMS and maximum height (Mh; see Table 1).

Table 1: Average roughness, root mean square and maximum height based on AFM.

Archwire	Variable	Sterilisation method	Values mean ± SD	F Statistic[a] (*df*)	*p*-value[a]
Nickel titanium (NiTi)	Ra (μm)	Control	2.08 ± 0.03	215.58 (3, 8)	0.001[b]
		Oven	1.54 ± 0.03		
		Autoclave	2.22 ± 0.05		
		2% Glutaraldehyde	1.98 ± 0.04		
	RMS (μm)	Control	3.44 ± 0.03	1338.44 (3, 8)	0.001[b]
		Oven	1.96 ± 0.04		
		Autoclave	2.92 ± 0.03		
		2% Glutaraldehyde	2.89 ± 0.03		
	Mh (μm)	Control	29.24 ± 0.53	2353.630 (3, 8)	0.001[b]
		Oven	11.47 ± 0.03		
		Autoclave	16.84 ± 0.04		
		2% Glutaraldehyde	19.91 ± 0.04		
Stainless steel (SS)	Ra (μm)	Control	0.98 ± 0.04	134.35 (3, 8)	0.001[b]
		Oven	1.18 ± 0.03		
		Autoclave	0.73 ± 0.06		
		2% Glutaraldehyde	1.44 ± 0.05		
	RMS (μm)	Control	1.44 ± 0.04	4.23 (3, 8)	0.046[b]
		Oven	1.44 ± 0.03		
		Autoclave	0.96 ± 0.03		
		2% Glutaraldehyde	1.72 ± 0.53		
	Mh (μm)	Control	8.10 ± 0.96	3.83 (3, 8)	0.057
		Oven	7.47 ± 0.54		
		Autoclave	6.72 ± 0.25		
		2% Glutaraldehyde	8.07 ± 0.21		

Notes: a = One-way ANOVA test; b = All 3 pairs of mean scores are significantly different by post-hoc test (Scheffe procedure)

The NiTi archwire sterilised with the oven had a significantly lower Ra value (1.54 ± 0.03, $p < 0.001$) than the control (2.08 ± 0.03) and NiTi archwires sterilised with the autoclave (2.22 ± 0.05) and 2% glutaraldehyde (1.98 ± 0.04). Concurrently, the oven-sterilised NiTi archwire had a significantly lower RMS value (1.96 ± 0.04, $p < 0.001$) than the control NiTi archwires (3.44 ± 0.03). No significant difference in RMS value was observed between the NiTi archwires sterilised with the autoclave (2.92 ± 0.03) and 2% glutaraldehyde (2.89 ± 0.03) and those sterilised with the oven. The Mh values for oven-sterilised NiTi archwires (11.47 ± 0.03, $p < 0.001$) were significantly lower than those of the control (29.24 ± 0.53), autoclave-sterilised (16.84 ± 0.04) and 2% glutaraldehyde-sterilised NiTi archwires (19.91 ± 0.04).

SS archwires subjected to autoclave sterilisation had significantly lower Ra value (0.73 ± 0.01, $p < 0.001$) than the oven-sterilised SS archwires (1.18 ± 0.03) and 2% glutaraldehyde-sterilised SS archwires (1.44 ± 0.05). The SS archwires sterilised with 2% glutaraldehyde had significantly higher Ra values (1.44 ± 0.05, $p < 0.001$) than the unsterilised SS archwires (0.98 ± 0.04). Similarly, the SS archwires sterilised with the autoclave had lower RMS values (0.96 ± 0.03, $p < 0.001$) than the control (1.44 ± 0.04), and SS archwires sterilised with the oven (1.44 ± 0.03) and 2% glutaraldehyde (1.72 ± 0.53). Nevertheless, no significant differences ($p > 0.05$) in Mh value were observed among the oven-sterilised SS archwires (7.47 ± 0.54), autoclave-sterilised SS archwires (6.72 ± 0.25) and 2% glutaraldehyde-sterilised SS archwires (8.07 ± 0.21) and the control (8.10 ± 0.96).

Machining and manufacturing processes determine the surface roughness of as-received or unsterilised control orthodontic archwires.[24] Amina and co-worker assessed the surface roughness of unsterilised NiTi and SS archwires through SEM profilometry;[24] their results showed that the NiTi archwires had higher profilometric surface roughness than the SS archwires, consistent with those of the current study, which showed that the Ra, RMS and Mh values of the NiTi archwires were higher than those of the SS archwires.

Autoclave sterilisation with moist heat is prevalent in the field of dentistry because it can cause irreversible damage to enzymes and proteins in microorganisms.[3] In the current study, autoclave sterilisation rendered the surface roughness of NiTi archwires higher than that of the untreated NiTi archwires and oven-sterilised NiTi archwires and 2% glutaraldehyde-sterilised NiTi archwires. However, this finding cannot be generalised because manufacturers use different methods to produce archwires. Furthermore, the current study utilised archwires from the same manufacturer, and 0.016-inch NiTi archwires were considered thick, which are mostly used in teeth alignment and commonly recycled because they are expensive.[9] SS archwires are prevalently used in orthodontics because they are reliable, cheaper than NiTi, compatible with different materials and corrosion resistant.[25] In this study, SS archwires subjected to cold sterilisation with 2% acidic glutaraldehyde and 10 h of incubation had significantly higher surface roughness than those sterilised with other methods. Clinicians may face difficulties when using cold sterilisation because many factors influence the depth and adequacy of the process, including immersion time, replacement of solutions at certain intervals and the oxidation or corrosion of SS materials or archwires soaked in disinfectant solutions, although 10 h of incubation period is deemed suitable for cold sterilisation.[26]

The 3D images obtained from AFM illustrate variations in the surface structure parameters of the NiTi (see Figure 2) and SS (see Figure 3) orthodontic wires.

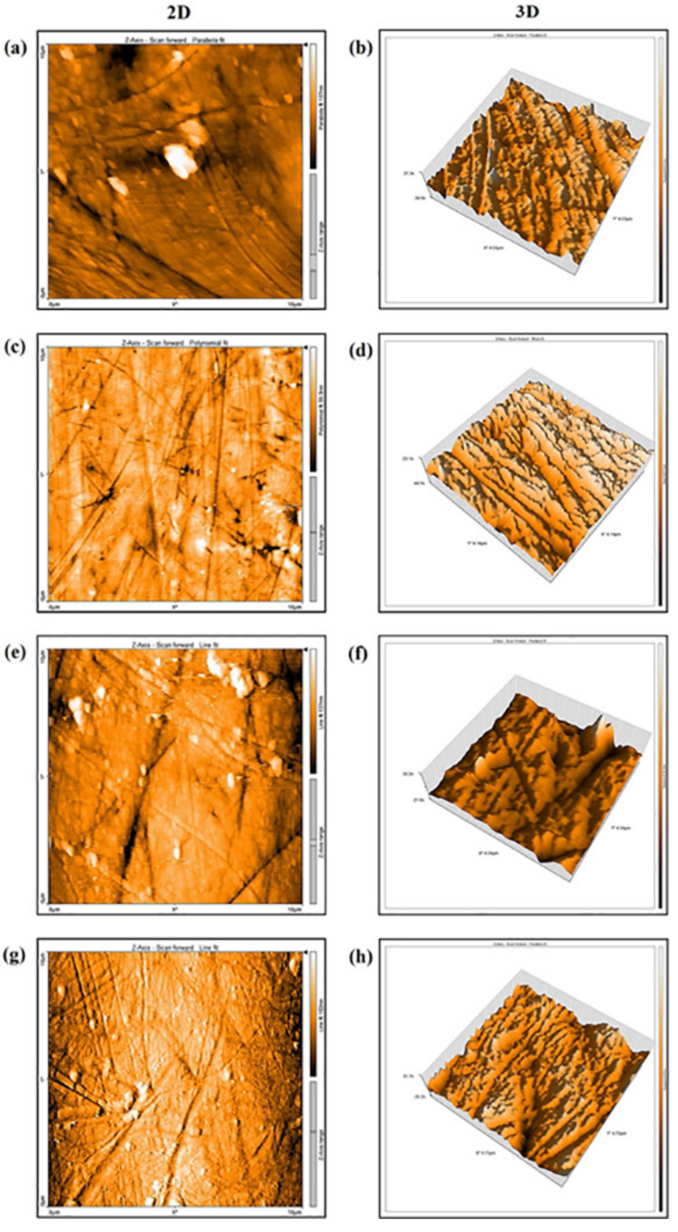

Figure 2: AFM 2D and 3D images of NiTi archwires subjected to different sterilisation methods. (a, b) Control, (c, d) oven sterilised, (e, f) autoclave sterilised and (g, h) 2% glutaraldehyde sterilised.

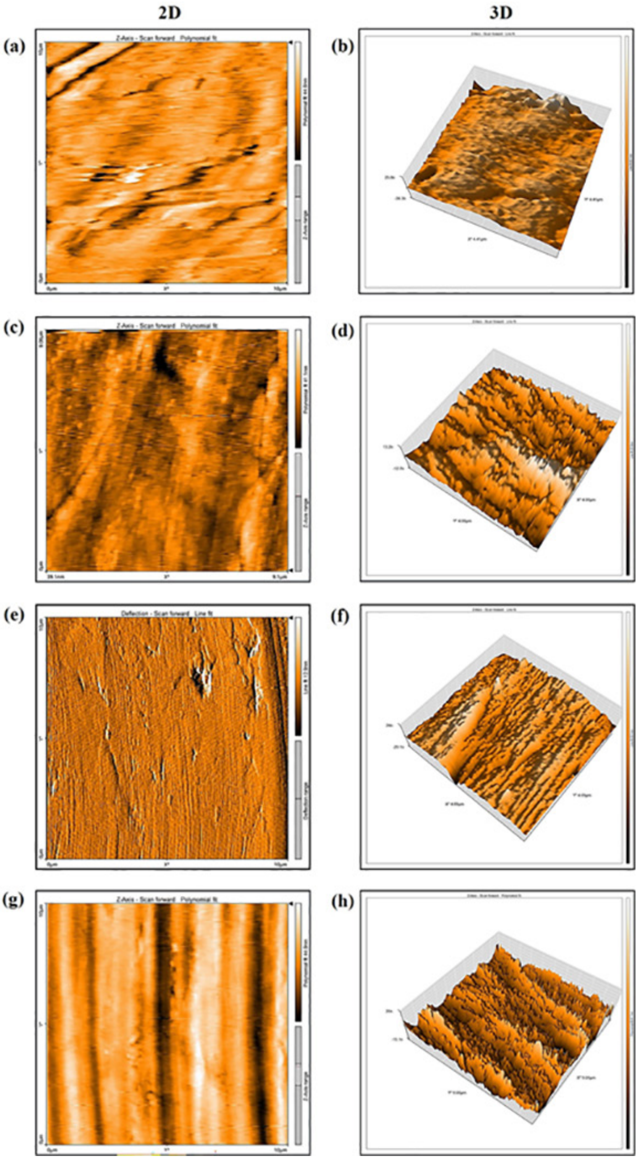

Figure 3: AFM 2D and 3D images of SS archwires subjected to different sterilisation methods. (a, b) Control, (c, d) oven sterilised, (e, f) autoclave sterilised and (g, h) 2% glutaraldehyde sterilised.

No noticeable variations in surface structure parameters were observed after sterilisation with different techniques. After steam autoclave sterilisation, the NiTi orthodontic archwire displayed an increase in surface roughness, and a uniform distribution was observed throughout the material. The surface roughness of NiTi archwires depends on grain arrangement and crystallisation that occurs when they are pulled through a diamond mould during their fabrication, and steam sterilisation may lead to the realignment of crystal grains.[27] The control SS archwire displayed a consistently smooth and even surface texture compared with the sterilised SS archwires. These results are consistent with those of a previous study, which reported that SS archwires had smoother surfaces than NiTi archwires after sterilisation with a steam autoclave.[4] However, SS archwires tend to show increase in surface roughness after clinical use, and autoclave sterilisation did not cause evident change in the archwires' characteristics.[28] An increase in surface roughness can cause an increase in friction between an archwire and bracket, affecting the efficiency during sliding mechanics and promoting corrosion.[12] These findings open new opportunities for finding archwire-coating materials suitable for clinical applications.

3.2 Surface Characteristics of Sterilised Orthodontic Archwires

The surface features of the NiTi and SS archwires without sterilisation (control) and those sterilised with the oven, autoclave and 2% glutaraldehyde were explored through SEM at ×2500 magnification. The surface topography of NiTi archwires is shown in Figure 4.

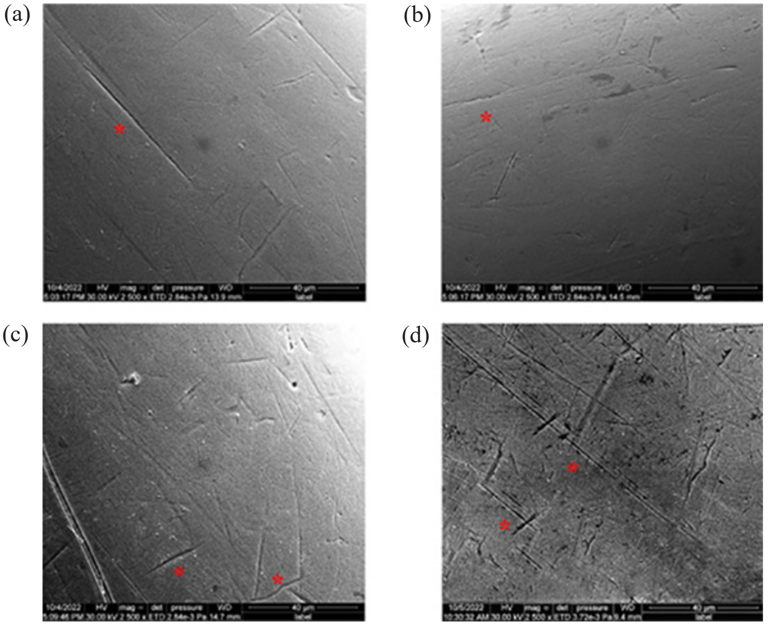

Figure 4: SEM images of NiTi archwire at ×2500 magnification with grooves (*). (a) Control, (b) oven sterilised, (c) autoclave sterilised and (d) 2% glutaraldehyde sterilised.

The control NiTi archwire had a smooth surface with visible lines and grooves (Figure 4[a]). After sterilisation, all the samples exhibited irregularities. NiTi archwires sterilised with hot air in the oven showed slight surface roughness with striations and almost had similar features to the control archwire (Figure 4[b]). The autoclave-sterilised archwires showed uneven surfaces with prominent striations and grooves (Figure 4[c]). Archwires exposed to 2% glutaraldehyde chemical sterilisation had rougher surfaces with multiple dispersed pores and had noticeable irregularities, including groove marks, small pits and dark spots, which were randomly distributed (Figure 4[d]). Moreover, 10 h of cold sterilisation with 2% acidic glutaraldehyde resulted in considerable changes in surface characteristics. This result is consistent with the results of Kapila et al.[29] In their study, NiTi archwires subjected to 10 h of cold sterilisation with 2% acidic glutaraldehyde caused substantial pitting, corrosion and changes in loading and unloading characteristics.[29]

Grosgogeat and colleagues demonstrated that autoclave sterilisation does not considerably modify the tribological properties of NiTi wires.[30] They also demonstrated that the surface roughness of NiTi archwires is not a determining parameter of tribological behaviour, adhesion, abrasion and surface structure; thus, they recommend the sterilisation of archwires prior to usage on patients.[30] Furthermore, Suresh and Kalidass showed that NiTi archwires exhibited small pores after autoclave sterilisation through SEM and AFM and the pores did not affect three-point bending load deflection behaviour, demonstrating that sterilisation is important to patients' safety.[16] An important parameter modulating frictional response is difference in hardness between a wire and bracket, and the relationship between the surface topography and mechanical properties of different archwires should be evaluated in future research.

The morphological features of the SS orthodontic archwires were examined through SEM analyses below at ×2500 magnification (see Figure 5).

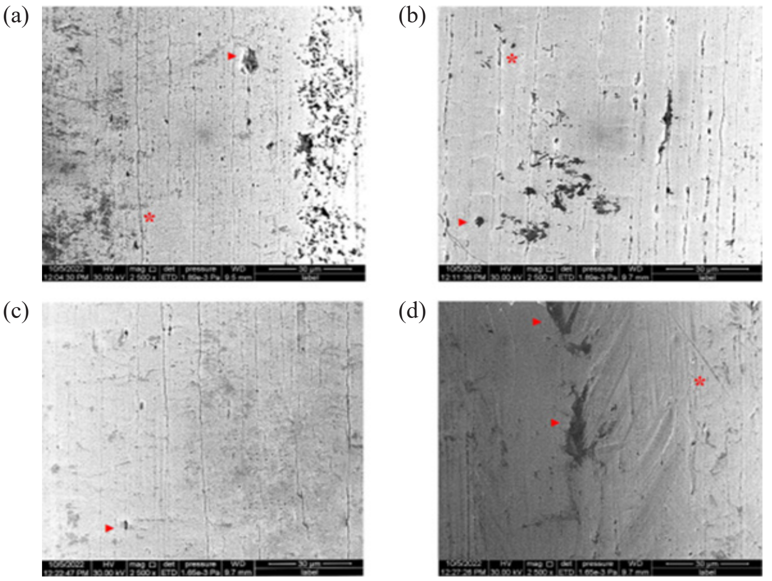

Figure 5: SEM images of stainless steel (SS) archwire at ×2500 magnification with pits (‣) and grooves (*). (a) Control, (b) oven sterilised, (c) autoclave sterilised and (d) 2% glutaraldehyde sterilised.

Surface regularities and parallel striations increased in the SS archwires. The SS archwires in the control group showed surfaces with striations parallel to the long axis and a pitting pattern (see Figure 5[a]). The pitting roughness of the SS archwires' surface topography showed a considerable increase after oven and 2% glutaraldehyde sterilisation. Oven sterilisation increased the number of surface regularities and parallel striations (Figure 5[b]), and autoclave-sterilised SS archwires (Figure 5[c]) exhibited small pores with uniform striations. The archwires sterilised with 2% glutaraldehyde showed scratches that were not parallel to the long axes of the archwires (Figure 5[d]). The SS archwires subjected to steam autoclave sterilisation exhibited smoother surfaces than those sterilised with the other methods. Cold sterilisation is widely used, although current guidelines recommend the use of steam autoclaving, which is rapid and uses moist heat.[31] SS archwires are regularly used because they are affordable to patients and can be recycled. Many recent studies have utilised SS archwires. The mechanical properties of SS archwires, such as modulus elasticity and stiffness, are reduced after steam autoclaving,[31] but this characteristic was not assessed in the current study.

EDS was used in analysing element composition in weight percentages (wt.%) present on the NiTi and SS archwires' surfaces after different sterilisation procedures (see Table 2).

Table 2: Elemental composition of NiTi and SS archwires according to EDS analysis.

Archwire	Group	Composition of element based on weight percentages (wt.%)												
		C	Si	Ni	Ti	Cr	Cu	Co	Fe	Ag	Mn	V	O	Total
NiTi	Control	10.5	0.3	48.5	40.5	–	–	0.2	–	–	–	–	–	100.0
	Oven	10.7	0.3	49.0	39.8	0.1	0.1	–	–	–	–	–	–	100.0
	Autoclave	13.4	0.3	46.8	39.2	–	0.2	0.1	–	–	–	–	–	100.0
	2% glutaraldehyde	14.4	0.3	46.3	38.9	–	0.1	–	–	–	–	–	–	100.0
SS	Control	11.1	0.6	7.1	–	16.8	–	–	62.6	0.3	1.4	0.1	–	100.0
	Oven	9.0	0.5	7.2	–	16.8	–	–	64.2	–	2.1	0.2	–	100.0
	Autoclave	9.3	0.6	7.2	–	17.2	–	–	63.3	0.1	1.8	0.1	0.4	100.0
	2% glutaraldehyde	7.4	0.5	7.5	–	17.1	–	–	65.7	0.1	1.6	0.1	–	100.0

Seven elements were detected on the surfaces of the NiTi archwires in the control group and those subjected to the three methods of sterilisation. Ni and Ti had the highest content in the NiTi archwires. The C content after 2% glutaraldehyde sterilisation was the highest (14.4%). The oven-sterilised NiTi archwires showed the highest Ni content (49.0%), and the untreated NiTi archwires had the highest titanium (Ti) content (40.5%). Chromium (Cr) at negligible amount (0.1%) was detected on the NiTi archwires after oven sterilisation. Copper was present in sterilised NiTi archwires' surfaces, but none was in the control. Cobalt was present in the control and autoclave-sterilised NiTi archwires.

For SS archwires, the highest element found on the surfaces was iron (Fe), ranging from 62.6% for the control archwires to 65.7% for the SS archwires sterilised with 2% glutaraldehyde sterilisation. Cr content increased from 16.8% (control) to 17.2% (SS archwires after autoclave sterilisation). C content was 11.1% in the control SS archwires and 7.4% in the SS archwires sterilised with 2% glutaraldehyde. Other elements, such as Ni, manganese (Mn), Si and vanadium (V) were also detected in the SS archwires.

EDS analysis provides insight into the chemical composition of NiTi archwires in the control group and those subjected to different sterilisation methods. The results indicated that the NiTi orthodontic archwires were composed of nearly equal proportions of Ni and Ti, and only slight variation in chemical makeup was observed before and after sterilisation. NiTi archwires have a consistent chemical composition, and the SE NiTi archwires have shape memory that can withstand temperatures of up to 482°C. This super elasticity allows for reversible phase transformation from austenitic body–centred structure to martensitic monolithic structure.[32] However, prolonged exposure to temperatures beyond 500°C may lead to the precipitation of Ti crystals on their surfaces.[32] Steam-sterilised NiTi archwires contain nearly equivalent Ni and Ti: 43%–44% Ni and 54%–55% Ti. The amount of cobalt ranges from 0.1% to 3% as reported by Petrov et al., which is consistent with the amount reported in the current study.[6] C was detected on the surfaces of all the NiTi archwires. Through Auger electron spectroscopy, Lepojević and co-worker showed that C may have originated from residual contamination in the atmosphere.[33]

No significant difference in chemical composition was found between the control SS archwires and all the sterilised SS archwires. The SS archwires' composition was mainly composed of Fe, Ni, Cr and C and depended on manufacturer's preferences. Changes in the amount of Fe was slightly higher (65.7%) in the SS archwires sterilised using 2% glutaraldehyde than in the control (62.6%), oven-sterilised (64.2%) and autoclave-sterilised SS archwires (63.3%). The Fe level in the current study was lower than the Fe levels reported by Arora et al., who revealed through EDS that as-received SS archwires (control) had higher Fe content in wt.% (73.43%) than clinically used steam autoclave–sterilised SS archwires (69.32%).[34] The presence of oxygen in SS archwires after autoclave sterilisation may be due to aqueous environments, which cause the formation of oxide layers because water is used to generate steam in steam autoclaving.[34]

Overall, the Ni content in the NiTi and SS wires was within normal limits. The composition of the SS archwires comprised 18% Cr and 8% Ni, whereas the NiTi archwires contained approximately 50% Ni in wt.%. The results of qualitative elemental analysis and quantitative elemental analysis through energy-dispersive X-ray (EDX) showed that Ni and Ti were the main elements in the examined archwires. Orthodontic archwires are regulated by International Standard Organisation (ISO) 15841:2014, and their use is not obligatory because all manufacturers are allowed to devise their archwire specifications during manufacturing. Thus, variations may occur among the batches of archwires produced by the same manufacturer.[34] In the present study, the results showed that sterilisation and surface treatment did not cause considerable changes in the tribological properties of the NiTi and SS archwires. The increased roughness was due to sterilisation, which eliminated debris or traces of grease that may have remained on the wires after production; thus, the true surface roughness of as-received wires is difficult to observe.[35]

3.3 X-ray Diffraction

Different XRD experiments were performed to complement the SEM measurements for the identification of the phases in the microstructure of the NiTi and SS archwires. The presence or absence of martensitic and austenitic phases was determined through XRD analysis before and after sterilisation. Studies on changes in NiTi and SS archwires phases after sterilisation using XRD are currently limited; hence, the current study aimed to report these changes.

The XRD analysis of the as-received NiTi archwires (control group) and sterilised ones was conducted at room temperature (Figure 6[a]). The results showed a diffractogram with three peaks (100, 200 and 211) at diffraction angles of 10°–90°, highlighting that the NiTi alloy with an austenite crystal structure system was cubic at 42°–43°. These results are consistent with those reported by Stoyanova–Ivanova et al., who highlighted that steam autoclaving at high temperatures did not change the NiTi austenite structure, which showed an ordered base-centred cubic structure at 2θ of 42.42° compared with the as-received archwires.[36] Moreover, Ilievska et al. showed that the XRD peaks of

NiTi orthodontic alloys were extremely similar in visual appearance to those obtained with conventional XRD.[37] They also showed that the XRD peaks of the NiTi orthodontic archwires were typically austenite-type structures.[37]

XRD analysis was performed on as-received SS archwires (control group) and sterilised SS archwires (Figure 6[b]). The patterns of diffraction demonstrated a standard cubic structure with three peaks (111, 200 and 220) at diffraction angles of 10°–90° and lattice in austenite phase at room temperature, indicating that the crystal system was cubic for the SS archwires. Many studies compared SS archwires normally presenting two main behaviours (standard temper or resilient temper) during heat treatment, steam autoclaving and cold sterilisation because the archwires' fundamental aspects related to metallurgical structure and mechanical properties remain unresolved.[6,38,39]

SS archwires with austenitic phase are metastable, but some conditions, such as extreme hot and cold temperatures, can decompose this phase into a martensitic phase, which is less stable and increases the susceptibility of SS archwires to corrosion.[38] SS archwires are made from American Iron and Steel Institute types 302 and 304 austenitic SS, and have either single-phase γ or double phase $\gamma + \alpha$ structures depending on C composition and temperature during manufacturing; stabilised Fe content in SS archwires with 18% Cr and 8% Ni is related to C composition in weight percentages (wt.%).[38] In the current study, the SS archwires' Ni content was 7.1%–7.5%, whereas its Cr content was 16.8%–17.2% which is within the acceptable range for orthodontic clinical usage.

Given that SS archwires are prone to corrosion, many studies have coated SS archwires with nanoparticles, such as titanium dioxide, carbon nitride and titanium nitride, which improve the corrosion behaviour of SS archwires; however, frictional behaviour over time must be investigated, especially when the archwires are used on patients and exposed to various temperatures in the oral cavity.[40,41]

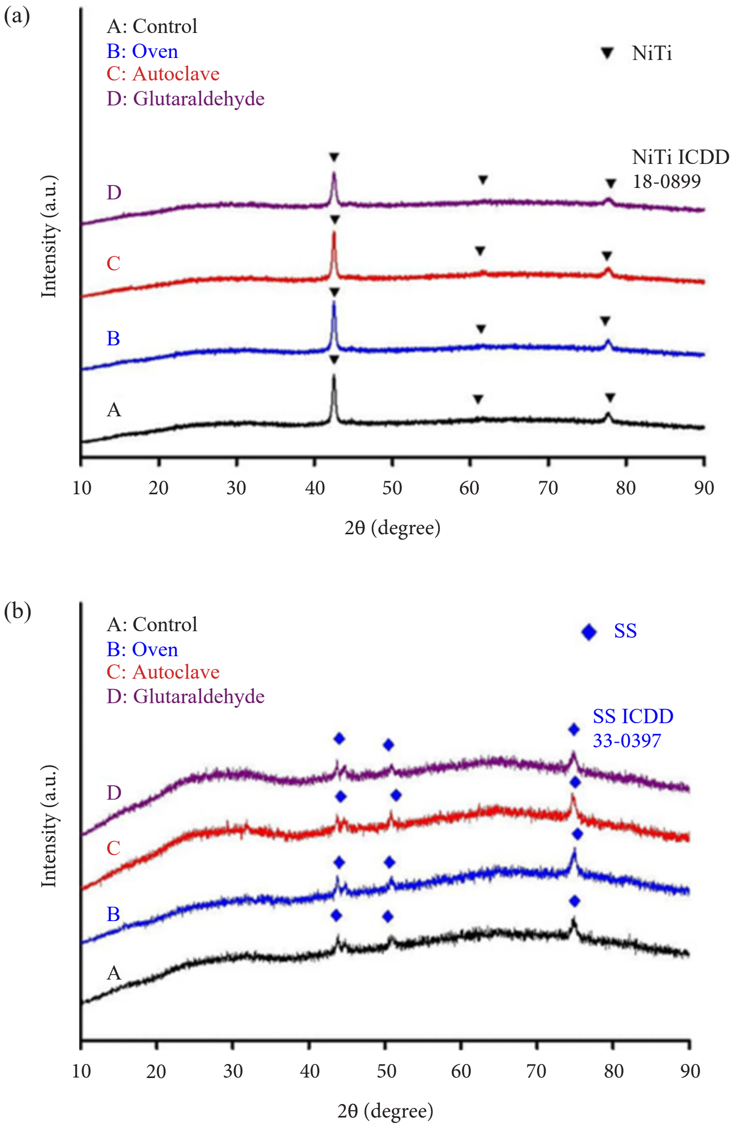

Figure 6: XRD diffractograms of the NiTi and SS archwires subjected to different sterilisation methods: (a) NiTi and (b) SS.

This study has some limitations because NiTi and SS archwires were obtained from the same manufacturer and the sample size with triplicates for each test was small. Thus, future studies should adopt archwires from different manufacturers. Nevertheless, the findings of the current study showed that sterilisation did not negatively affect the surface characteristics of NiTi and SS archwires, and presence of coatings may prevent the corrosion of archwires in the oral environment.

4. CONCLUSION

This study examined the surface characteristics of NiTi and SS orthodontic archwires subjected to three types of sterilisations, namely, heat sterilisation with an oven, steam and rapid sterilisation with an autoclave and cold sterilisation with 2% acidic glutaraldehyde. Sterilisation was performed in accordance with the recommended guidelines for orthodontic metallic archwires. The SEM results showed that the surface characteristics of the sterilised NiTi and SS archwires did not exhibit considerable changes compared with the untreated archwires. The SS archwires in the control group had smoother surfaces than the NiTi orthodontic archwires before sterilisation, and EDS analysis showed that sterilisation did not result in noticeable changes in the chemical composition of the NiTi and SS orthodontic archwires. In addition, Ni and Ti were the main elements in the NiTi archwires, whereas Fe and Cr were the main elements of the SS archwires.

AFM analysis provides enhanced surface topography and can be used in identifying changes in orthodontic archwires' surfaces before and after sterilisation and is thus recommended for future studies related to coated or uncoated archwires after sterilisation. No significant differences in the roughness of NiTi and SS archwires were observed after sterilisation. XRD analysis showed no significant changes in the crystalline phases on the NiTi and SS archwires subjected to oven, autoclave and 2% glutaraldehyde sterilisation compared with the control untreated archwires.

Ovens, autoclaves and chemical sterilisation solutions can be used to sterilise orthodontic wires because they do not adversely affect the surface roughness of the wires.

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