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# Impact of recycling on the mechanical properties of nickel-titanium alloy wires and the efficacy of their reuse after cold sterilization

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## Abstract:

**OBJECTIVE:** This study aimed to assess the feasibility of reusing nickel–titanium (NiTi) alloy wires after 6 weeks of intraoral use by evaluating the changes in the load-deflection properties and surface characterization of these alloy wires after cold sterilization by immersion in 2% of acidic glutaraldehyde for 10 h.

**MATERIAL AND METHODS:** Twenty wires each in three groups of G1-as-received wires (ARW), G2-unsterilized used wires, and G3-sterilized used wires (SUW) were tested by the three-point bending test and scanning electron microscopy (SEM). The data were subjected to statistics, one-way analysis of variance, and Bonferroni posthoc test for comparison.

**RESULTS:** Recycling of NiTi wires produced statistically insignificant changes in both the loading and unloading properties of the wires. The forces needed to twist the used wires, that is, G2-(UUW) and G3-(SUW) were lower than G1-(ARW), suggesting lowering of the stiffness of the wires. Superelasticity is well-maintained by G2-(UUW) and G3-(SUW) although there is an insignificant lowering of the forces exerted by them during loading and unloading. SEM demonstrated no increase in the pitting of surfaces in both G2-(UUW) and G3-(SUW); multiple areas were seen to be more smoothed over G2-(UUW) and G3-(SUW) NiTi wires surfaces.

**CONCLUSION:** The findings of this study support the reuse of NiTi wires after 6 weeks of use in oral conditions followed by cold sterilization by immersion in 2% acidic glutaraldehyde for 10 h.

## Keywords:

Bending test, cold sterilization, nickel-titanium alloy wires, recycling, superelasticity

## Introduction

Nickel–titanium (NiTi) alloy wires gained popularity because of their properties like superelasticity and shape memory. However, because of the higher cost value, more than 50% of the orthodontists recycle these wires for economic reasons.<sup>[1,2]</sup> To eliminate the potential health hazards to patients on whom these recycled archwires are used, effective sterilization methods

must be used.<sup>[3-5]</sup> Approximately 80% of these orthodontists use chemical solutions, that is, a cold method for disinfecting or sterilizing these wires. The most popular disinfectants and sterilants, authorized by the American Dental Association, include 2% glutaraldehyde and chlorine dioxide for the 2% acidic glutaraldehyde (Banicide) sterilization time is 10 h without any dilution. Most of the disinfectants and sterilants are reportedly corrosive and attack the metallic substances that are immersed in them.<sup>[1]</sup>

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To reuse NiTi wires following cold sterilization treatment in patients, these must be assessed for selected properties like stiffness, strength, and surface characteristics. Few studies exist on the effects of cold sterilization with chemical solutions on NiTi wires. This study was conducted to assess the feasibility of reusing NiTi alloy wires by evaluating the changes in mechanical properties and their surface characteristics after cold sterilization by immersion in 2% acidic glutaraldehyde for 10 h.

## Methods

Sixty 0.016 NiTi alloy archwires (Nitinol Superelastic Wire, 3M Unitek, CA) were divided into three groups of 20 samples each. Twenty as-received wires (ARW) served as control (G1). The remaining 40 wires were placed intraorally for a period of 6 weeks in patients undergoing orthodontic treatment. After 6 weeks, these wires were taken out and cleaned with 70% isopropyl alcohol for the removal of any debris. Out of these 40 wires, 20 unsterilized used wires (UW) formed the second group (G2). A third group (G3) comprised the remaining 20 sterilized used wires (SUW) that were sterilized using 2% acidic glutaraldehyde for a duration of 10 h. A three-point bending test was conducted to ascertain the load-deflection properties of the nickel-titanium archwires.<sup>[6]</sup> A typodont set with brackets bonded on it using adhesive was used as a jig [Figure 1]. The test wires were secured to the brackets with the elastomeric modules. The first premolar was removed from the typodont set. The distance kept between the midaxes of brackets from the canine and the second premolar was 14 mm apart. A bracket bonded to the metallic rod, which gets attached to the load cell [Figure 2], was used to apply force to deflect the wire section between the canine and premolar brackets. The jig is attached to the crosshead of an Instron machine (Instron Corp., Canton Mass) with 50 kg force on load cell [Figure 3]. The speed of the crosshead of the testing machine was set at 1 mm/min for a total of 2 mm deflection for the loading of the wire. The crosshead was then reversed and the wire was unloaded. The forces required to deflect the wires for 0.2 mm intervals during loading and unloading were recorded and plotted for displacement on the X-Y recorder. After every test run, the next wire to be tested was relegated and the entire procedure was repeated. A scanning electron microscope (SEM, ZEISS EVO 50) was used to arbitrarily choose and examine six different segments of the wire specimens, two specimens from each group. Representative SEM images of the wire specimens were studied at a magnification of 1000× to expose any changes in the surface texture of the NiTi wires after using 2% acidic glutaraldehyde. Ethical approval was obtained from the institutional ethical



Figure 1: Typodont set used as a jig



Figure 2: Metallic rod with bracket

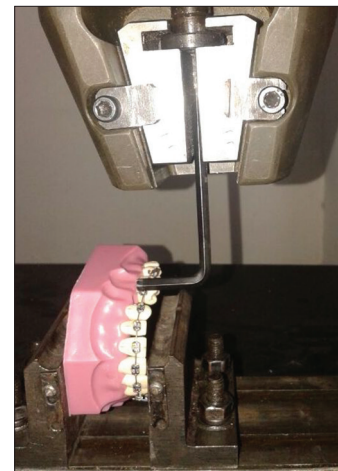


Figure 3: Universal testing machine crosshead with jig attached for three-point bending test

committee (TMDC/18/34-456). The Statistical Package for the Social Sciences software (version 21) and Epi Info version 3.0 were used for the statistical analysis.

## Results

The values obtained for G1, G2, and G3 wires during loading and unloading were tabulated, and the average mean at each interval was calculated [Figures 4 and 5].

Table 1 shows the comparison of the mean peak load at 2 mm deflection between the three groups done using a one-way analysis of variance. Test results revealed a highly significant difference ( $P < 0.01$ ) between the three groups. G1-(ARW) showed the highest mean peak load value of  $304.60 \pm 38.15$  g. G2-(UUW) showed the mean peak load value of  $254.20 \pm 42.51$  g, which was the least of all the three tested groups. The mean peak load value of G3 was  $268.90 \pm 44.11$  g.

Table 2 for intergroup comparison revealed a highly significant difference ( $P < 0.01$ ) in the mean peak load values between the G1-(ARW) and G2-(UUW). The difference in the mean peak load between the G1-(ARW) and G3-(SUW) is also significant ( $P < 0.05$ ), but the mean peak load difference value between the G2-(UUW) and G3-(SUW) is nonsignificant ( $P > 0.05$ ).

Table 3 represents the mean force lost during unloading of the wires in the groups G1-(ARW), G2-(UUW), and G3-(SUW), when the wire deflection was decreased by 0.6 mm, that is, from 1.6 mm to 1.0 mm. Mean values of the force lost during unloading for G1-(ARW), G2-(UUW), and G3-(SUW) were  $21.50 \pm 17.30$  g,  $14.75 \pm 18.39$  g, and  $15.70 \pm 15.22$  g, respectively. A comparison of mean force lost during unloading between G1-(ARW), G2-(UUW), and G3-(SUW) shows a nonsignificant difference among the three groups of wires.

**Table 1: Mean peak load (in grams) at 2 mm deflection**

	Mean	SD	Standard error	F	P
ARW (G1)	304.6	38.15	8.53	7.740	0.001**
UUW (G2)	254.2	42.51	9.50		
SUW (G3)	268.9	44.11	9.86		

\*\* $P < 0.01$  (highly significant). ARW: as-received wire; UUW: unsterilized used wire; SUW: sterilized used wire

**Table 2: Intergroup comparison of mean peak load (in grams) at 2 mm deflection**

(I) GPs	(J) Gps	Mean peak load	P
G1-(ARW)	G2-(UUW)	50.40	0.001**
G2-(UUW)	G3-(SUW)	14.70	0.808
G3-(SUW)	G1-(ARW)	35.70	0.027*

\* $P < 0.05$  (significant), \*\* $P < 0.01$  (highly significant)

**Table 3: Force (in grams) lost on unloading curve between the interval from 1.6 mm to 1.0 mm**

	Mean	SD	Standard error	F	P
ARW (G1)	21.50	17.3	3.87	0.922	0.404
UUW (G2)	14.75	18.39	4.11		
SUW (G3)	15.70	15.22	3.40		

Figure 6 graph for G1-(ARW) shows a nonlinear load-deflection curve. In the loading curve of G1-(ARW), the average mean force at 0.4 mm of deflection is 151.6 g at 1.0 mm of deflection force value is 234.6 g, thus, force value increased by 83 g (54.96%). During the unloading of the wire, the average mean force at 1.0

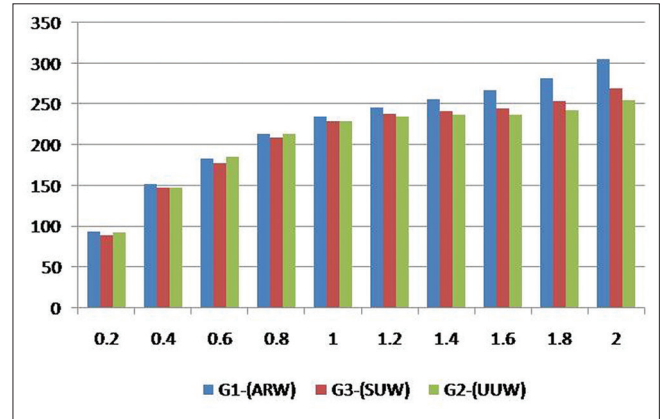


Figure 4: Average mean values of force (in grams) at an interval of 0.2 mm deflection during loading of wires

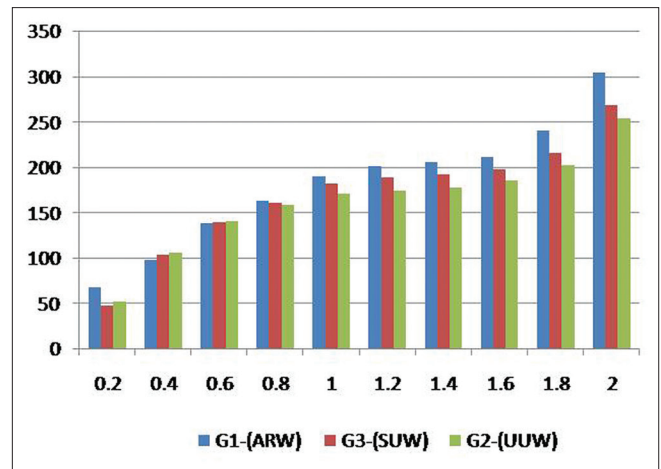


Figure 5: Average mean values of force (in grams) at an interval of 0.2 mm deflection during unloading of wires

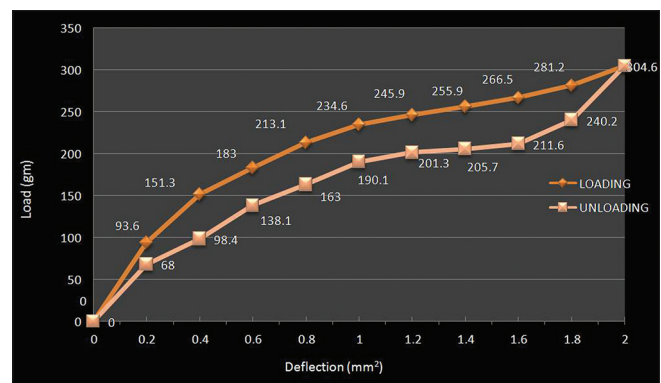


Figure 6: Graphical representation of force values (in grams) and their average means obtained at intervals of 0.2 mm deflection during loading and unloading of G1-as-received wires (ARW)

mm of deflection of the wire was 190.1 g, and then, the force value decreased by 91.7 g (48.23%) from 98.4 g at 0.4 mm of deflection.

Figure 7 for the G2-(UUW) shows a nonlinear load-deflection curve. The mean peak load value of G2-(UUW) is  $254.2 \pm 42.51$  g at 2 mm deflection. In the loading curve of G2-(UUW), the average mean force at 1 mm of deflection was 228.80 g, and the force value became 266.5 g at 1.6 mm of deflection, that is, it increased by 8.10 g (3.49%) while loading. During the unloading of the wire, the average mean force at 1.6 mm of deflection of the wire was 185.45 g, and it was 170.7 g at 1.0 mm interval of deflection curve, that is, it decreased by 14.75 g (7.97%). This shows that the wire shows superelastic property in this region. In the unloading curve of G2-(UUW), the average mean force at 0.4 mm of deflection was 144.6 g, and the force value became 228.8 g at 1.0 mm of deflection while loading. Thus, the force value increased by 82.2 g (56.07%) for further 0.6 mm increase in deflection. During unloading of the wire, the average mean force at 1.0 mm of deflection of the wire was 170.7 g, and then, the force value decreased by 64.5 g (37.78%) from 106.2 g at 0.4 mm of deflection.

Figure 8 representing G3-(SUW) shows a superelastic plateau during loading and unloading of the wire. In the loading curve, the average mean force at 1 mm of deflection was 229.20 g, and then, it increased by 15.20 g (6.63%) for further 0.6 mm increase in deflection (i.e., 244.40 g at 1.6 mm). During the unloading of the wire, the average mean force at 1.6 mm of deflection of the wire was 197.70 g, and then it, decreased by 15.70 g (7.94%) to 182.00 g at 1.0 mm interval of the deflection curve. In the loading curve of G2-(SUW), the average mean force at 0.4 mm of deflection was 146.50 g, and then, the force value increased by 82.70 g (56.45%) for a further 0.6 mm increase in deflection, that is, the force value became 229.20 g at 1.0 mm of deflection while loading. During the unloading 40 of the wire, the average mean force at 1.0 mm of deflection of the wire was 182.00 g, and then, the force value decreased by 78.90 g (43.35%) from 103.10 g at 0.4 mm of deflection.

Figure 9 shows that G1-(ARW), G2-(UUW), and G3-(SUW) NiTi wires demonstrate Pseudoplasticity, as the wires are displaced from 1 mm to 1.6 mm during loading of the wires, and pseudoelasticity, as the wires reverted from 1.6 mm to 1 mm deflection during unloading.

### Surface Topography

The SEM image of G1-(ARW) at 1000× magnification is shown in Figure 10a which reveals that the ARW shows many round or oval pitting and relatively wider

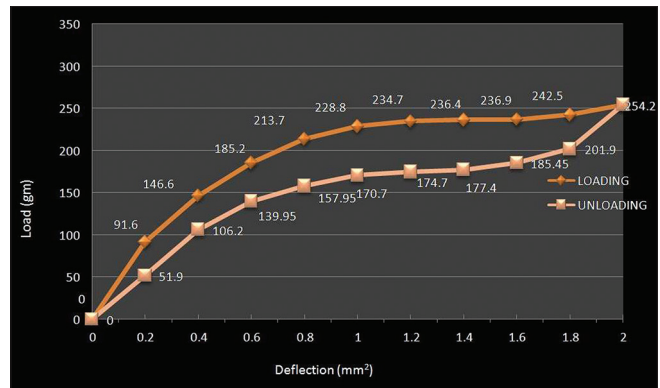


Figure 7: Graphical representation of force values (in grams) and their average means obtained at intervals of 0.2 mm deflection during loading and unloading of G2-sterilized used wires (UUW) wires

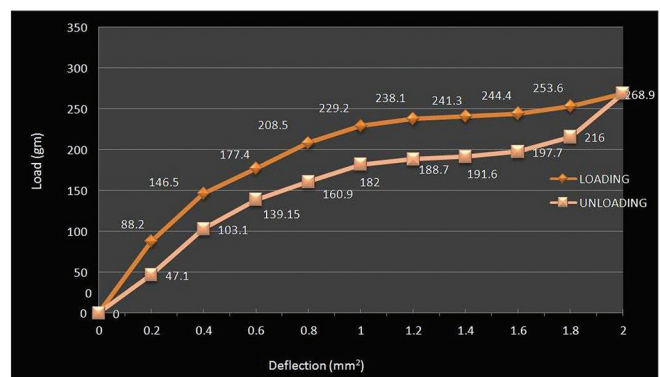


Figure 8: Graphical representation of force values (in grams) and their average means obtained at intervals of 0.2 mm deflection during loading and unloading of G3-sterilized used wires (SUW)

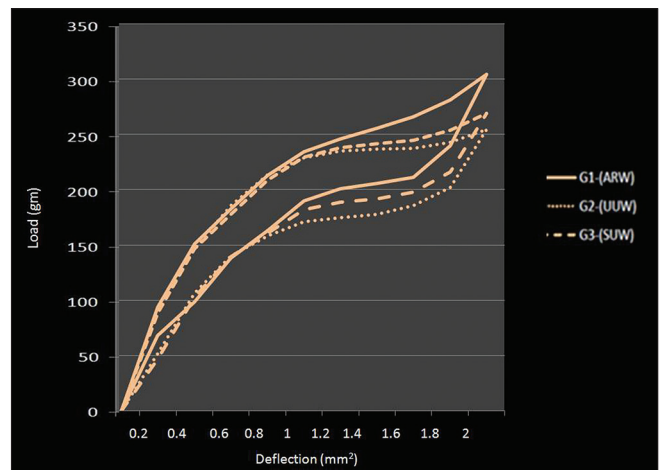


Figure 9: Graphical representation of the comparison of the load-deflection curves of G1-(ARW), G2-(UUW), and G3-(SUW)

depressions that must have been created during the manufacturing process of these wires. The SEM image of G2-(UUW) at 1000× magnification is shown in Figure 10b, which reveals a smoother surface of the wire than the ARWs. The presence of deep indentations was seen on the wire surface, which was because of the drawing

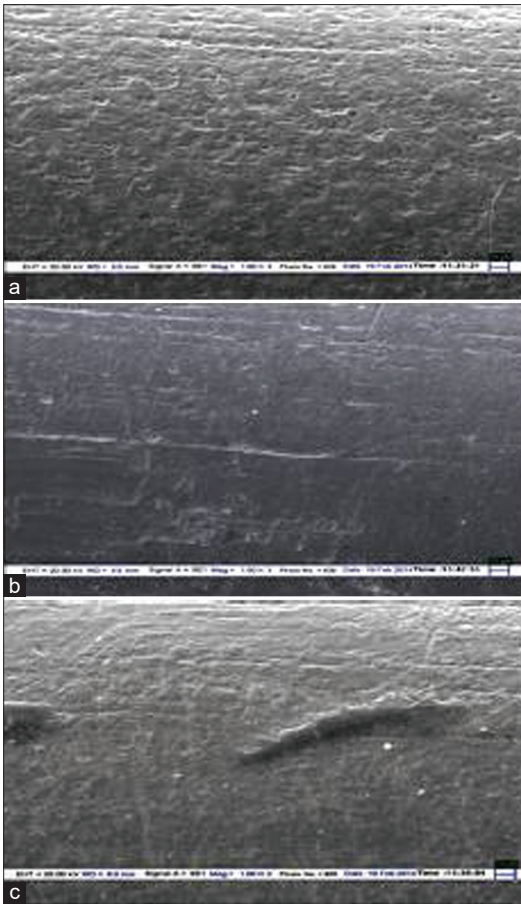


Figure 10: (a-c) SEM images of G1-(ARW), G2-(UUW), G3-(SUW) at 1000× magnification

process of the wire while the manufacturing process. SEM image of G3-(SUW) at 1000× magnification is shown in Figure 10c, revealing a smoother surface of wire as compared with the ARW group, which could be explained by the wear occurring between the wire and the bracket surfaces. Notches were present at certain places. There was a presence of certain prominences and depression which could be because of the defect in the manufacturing process.

## Discussion

Many studies have reported an intraoral deterioration of NiTi wires because of corrosion in the fluoride-rich environment.<sup>[4,7-9]</sup> The feasibility to use these wires after 4 to 6 weeks of intraoral use is a matter of concern. Dry heat, cold sterilization, and autoclaving of the used NiTi wires are the reported methods for sterilization.<sup>[10,11]</sup> Various studies have reported that the nitinol and titanium alloy wires can be heat sterilized without deteriorating their mechanical properties. Nickel–titanium wires undergo phase changes because of heat treatment which alters their properties.<sup>[4]</sup> Temperatures greater than 60°C increased the vulnerability of these wires to deform plastically and reduced their springiness.<sup>[8,10,11]</sup>

Intraoral use exposes the wire to physical stresses and oral conditions like thermodynamic changes and forces from mastication and occlusion. G2-(UUW) has been included in our study to understand these changes in the mechanical and surface characteristics of the used NiTi wires. In G3-(SUW), the wires were exposed to the effects of physical stresses in oral conditions as well as cold sterilization, which subjected it to corrosion attack from 2% glutaraldehyde and cold working, which can both alter its properties.

To demonstrate the differences between the first nitinol wire and the superelastic NiTi wires in 1986, a three-point bending test was introduced by Miura.<sup>[12]</sup> However, many other authors have also advocated that the archwire should be tested under restraint so that the wire is not free at both ends to simulate the clinical situation. In addition, higher force values during loading and unloading are obtained, as compared with previous methods.<sup>[13]</sup> Beyond 2 mm of deflections, permanent deformation starts to set in; thus, most of the studies use a range of 0.2 mm of deflections.<sup>[1,2,5,12]</sup> The loading portion of the graph simulates the activation of the wires; whereas, the unloading section of the graph denotes the deactivation of force, which causes tooth movement during clinical performance.<sup>[1-3,12]</sup>

The G1-(ARWs) were found to have significantly higher loading and unloading forces than G2 and G3 wires. The crystallographic behavior of the NiTi wires in our study resembles that of austenitic NiTi alloys when interpreted by the stress–strain graph. The initial linear loading curve represents a purely elastic deformation of the austenitic phase. The curve flattens to a nonlinear pattern at the same load (pseudoplasticity), where the martensitic transformation begins. Level of the plateau signifies the load exerted during the completion of martensitic transformation, which is lower for recycled wires. When the reverse transformation to the austenitic phase begins during the unloading, the graph again shows plateau (pseudoeasticity), at a particular load from where stress-induced martensitic structure exists. In the final part of the deactivation curve, the phase transformation from the martensitic to austenitic phase is completed [Figures 6–9]. The unusual nature of the superelastic material is that the loading curve differs from the unloading curve (hysteresis) is depicted by the load-deflection curves of the three groups.

The recycled NiTi wires (G2 and G3) exerted reduced forces while loading and unloading compared with G1 wires. There were statistically significant changes in loading and unloading forces in the interval between 0.6 mm and 1.0 mm during loading and 1.0–0.6 mm during unloading. This showed a reduction in the pseudoelastic

characteristics during unloading in the lower ranges of deflection in the recycled NiTi wires, which exhibits as work-hardening caused by the summative effects of masticatory forces and abrupt intraoral temperatures changes. Similar findings have been reported by other authors.<sup>[12,14]</sup> It suggests that recycled NiTi wires must be activated more frequently or earlier archwire changes must be exercised. The comparison of mean peak load at 2 mm deflection reveals a highly significant difference ( $P < 0.01$ ) among the tested groups. G1-(ARW) showed the highest mean peak load value of  $304.60 \pm 38.15$  g. Both G2-(UUW) and G3-(SUW) groups showed the decreased value of mean peak load and the difference was statistically significant. These findings are in accordance with many other studies.<sup>[1,14]</sup> They show that there is a reduction in loading and unloading forces, after the clinical use of 6 weeks (G2) and (G3). Kapila *et al.* have reported that the clinical use contributes more than the sterilization in causing the changes in the load-deflection characteristics.<sup>[1,5,15]</sup> According to Segner *et al.*, a plateau value of 0.5 mm is considered a good value.<sup>[15]</sup> The recycled NiTi wires (G2 and G3) have a clinical plateau of 0.6 mm length; thus, the recycled wire showed very well-maintained superelastic characteristics that are needed for clinical use. The finding of our study supports the recycling of NiTi wires, as these wires retain their desirable mechanical properties after cold sterilization with 2% acidic glutaraldehyde. However, the testing procedure used is a static environment, that is, thermal and dynamic changes, such as forces of mastication and occlusion were nonexistent.<sup>[12,13]</sup>

Many investigators have reported increased sensitivity of the recycled wires to corrosion; thus, surface characteristics were assessed with SEM to study the surface topography of the three groups.<sup>[9,12]</sup> SEM specimens were examined at 1000× magnifications. The images obtained for representative segments of recycled wires showed no signs of increased pitting. Instead, G2 and G3 wires demonstrated areas of smoothness and some surfaces of the wires were scored. This smoothing and scoring results from abrasion because of the sliding and rubbing of these wires within the bracket slot, as explained by previous investigators.<sup>[15-17]</sup> Some reports have concluded that in-vitro corrosion does not affect the physical properties of recycled NiTi wires.<sup>[18]</sup> However, nickel dissolution that occurs from corroded surfaces of NiTi wires could have adverse reactions in patients previously sensitized to nickel.<sup>[19]</sup> The findings of this study suggest the possibility of reuse of recycled NiTi wires after 6 weeks following cold sterilization using 2% acidic glutaraldehyde for 10 h; however, further research is needed to validate its clinical application.

## Conclusion

There is a reduction in stiffness exhibited by the recycled NiTi wires after 6 weeks of clinical use. The surface topography of the clinically exposed wires also shows no increase in pitting of the surface, indicating no sign of corrosion attack because of oral environment or sterilization procedure with 2% glutaraldehyde. The findings of this study support the reuse of NiTi wires after 6 weeks of use in oral conditions, followed by cold sterilization by immersion in 2% acidic glutaraldehyde for 10 h.

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## Conflicts of interest

There are no conflicts of interest.

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