Original Article

Evaluation of Nanomechanical Properties, Surface Roughness, and Color Stability of Esthetic Nickel-Titanium Orthodontic Archwires

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ABSTRACT

Objectives: The objective of the study is to evaluate the surface roughness, nanomechancial properties the color stability of three brands of coated (rhodium, epoxy, and Teflon) nickel-titanium (NiTi) esthetic archwires. **Materials and Methods:** Three brands of coated (rhodium, epoxy, and Teflon) esthetic NiTi archwires and three brands of uncoated (NiTi) archwires from the same manufactures were evaluated for the surface roughness, nanomechanical properties, and color stability. The specimens with 20 mm length (n = 5) were cut from the straight buccal segments of the coated and uncoated archwires. The specimens with 20 mm length (n = 10) were subjected to color measurement after immersion in a coffee staining solution. The color measurement was evaluated after 7, 14, 21, and 28 days after immersion in staining solution using color eye 7000 spectrophotometer. The experimental data were analyzed using descriptive statistics, analyses of variance, and Tukey's *post hoc* test. **Results:** Epoxy (1.517 ± 0.071) and rhodium (0.297 ± 0.015) coated archwires showed the highest and lower value of surface roughness. All the intergroup comparisons showed a significant difference ($P < 0.05$) in surface roughness except between rhodium and control group ($P = 0.998$). There were significant differences between control and the experimental groups for both nanohardness and elastic modulus was observed. All the three NiTi-coated esthetic archwires demonstrated trace" (extremely slight change) color changes as measured by the National Bureau of Standards units after 4 weeks of immersion. **Conclusion:** Surface roughness of rhodium-coated archwires was almost similar to that of uncoated wires. Whereas Teflon and epoxy coated archwires showed a significant difference in surface roughness compared to uncoated archwires. Uncoated archwires showed higher nanohardness values compared to the coated archwires. Teflon-coated archwires demonstrated significantly slight color change after 4 weeks of immersion in staining solution.

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INTRODUCTION

Patients undergoing orthodontic treatment are increasingly demanding for the better esthetic. This led to the introduction of orthodontic appliances that combine the esthetics with optimal performance.[1-3] In fixed orthodontic therapy, the introduction of esthetic orthodontic brackets partially solved the issue, but most of the orthodontic wire alloys are stainless steel, cobalt-chromium, beta-titanium, and nickel-titanium (NiTi).[4]

Metallic archwires are coated with colored polymers or inorganic materials to fulfill the growing esthetic needs of the orthodontic patients.[5] Materials used in esthetic

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coating are polymers such as synthetic fluorine-containing resin or epoxy resin or polytetrafluoroethylyene (PTFE Teflon), which simulates the tooth color.[6,7]

The process of coating the archwires involves surface treatment of the wire and the use of clean compressed air as a transport medium for the atomized PTFE particles to coat the wire. The entire setup is further heat treated in a chamber furnace. The mechanical properties of metallic archwires could be affected during the coating process and induce changes to their inner alloy core dimensions to compensate for the thickness of the coating layer.[8] A novel method of coating commercially available orthodontic wires by nanoceramics with sol-gel thin film dip coating technique has been found to be successful.[9] Moreover, coatings of the archwires varied in thickness. Archwires with uniformly thicker coatings demonstrated better properties than the thinner coated archwires.[10]

Previous studies have reported conflicting results about the esthetic-coated archwires. A study of coating adherence and sliding properties showed that the coating decreased friction between archwires and brackets[11,12] while some authors argued that the archwires are not durable, change in color with time and lose the coatings thereby exposing the underlying substrate metal. All these affects the area of surface contact, esthetics concerns, corrosion, and the biocompatibility of the archwires.[1,13]

Previous studies have evaluated the mechanical and physical properties of orthodontic archwires by employing various techniques. However, the data regarding the surface topography, color stability, and nanomechanical properties of currently marketed coated esthetic archwires is sparse. Hence, the present study aimed to evaluate and compare the surface roughness, nanomechanical properties, and color stability of three brands of uncoated and coated esthetic orthodontic archwires.

Materials and Methods

Ethical approval

This study was registered and ethical approval obtained from the research center of Riyadh Elm University. The study was assigned this registration number FPGRP/43534002/69.

Study design

This was an experimental study conducted in the laboratory. Three brands of coated esthetic (NiTi) archwires and three brands of uncoated archwires from the same company were evaluated in the present study. All the archwires were rectangular in cross-section and had the same cross-section size (0.17 × 0.25). The archwires with their brand name, type of coating, and manufacturer details are given below.

Coated archwires (study group)

1. Brand: Rabbit force NiTi
   - Coating: Epoxy
   - Manufacturer: Libral Traders, New Delhi, India.
2. Brand: Navy NiTi
   - Coating: Rhodium
   - Manufacturer: Libral Traders, New Delhi, India.
3. Brand: Tooth Tone.
   - Coating: Plastic
   - Manufacturer: Ortho Technology, FL, USA.

Uncoated archwires (control group)

4. Brand: Rabbit Force NiTi
   - Uncoated
   - Manufacturer: Libral Traders, New Delhi, India.
5. Brand: Navy NiTi
   - Uncoated
   - Manufacturer: Libral Traders, New Delhi, India.
6. Brand: Tru Flex
   - Uncoated
   - Manufacturer: Ortho Technology, FL, USA.

Evaluation of surface roughness

Specimen preparation and testing

Five specimens per each group of archwires ($n = 5$) were used for surface roughness evaluation. The sample size calculation was in accordance with previously reported studies for surface roughness evaluation.[11] The specimens were new in packaging, performed in arch forms, each sample was cut into 20 mm length from the straight buccal segments of the coated and uncoated archwires using orthodontic soft wire cutter. The cut wire was cleaned with distilled water to remove any surface impurities. The cleaned wire was dried using tissue paper and was kept ready for profilometry evaluation. A non-contact surface profilometer with a three dimensional (3D) optical feature (Bruker Contour GT, Tucson, AZ, USA) was used for surface evaluation [Figure 1]. The profilometer works on contact scanning white light interferometry (interferometer is an optical device that divides a beam of light exiting from a single source into two beams and then recombines them to create an interference pattern). The profilometer uses a nanolens atomic force microscopy module and has a fully automated turret and programmable X, Y, Z movements which is controlled by Vision 64 software (Bruker Corporation, San Jose, CA, USA). The vision 64 software transforms the high-resolution data into accurate 3D images. The wire specimens were secured on to the movable turret such that the 0.025 surface facing the light source of profilometer. The specimens were scanned in five random areas. The mean of the 5 measurement values corresponded to the surface roughness of that particular specimen (Ra).

Evaluation of nanomechanical properties

Specimen preparation and testing

The cleaned wire was dried using a tissue paper and was kept ready for nanoindentation. The nanomechanical properties (hardness and elastic modulus) were measured using nanoindenter (Bruker, Tucson, AZ, USA) equipped with a Berkovich diamond indenter [Figure 2]. The wire specimens were secured on to the movable turret such that the 0.025 surface facing is toward indenter. The test was performed in air under ambient temperature of 23°C and low noise conditions in a closed chamber. The indenter loading rate was 0.01 mN/s, and unloading rates were 0.02 mN/s. The resting period was 5 s at maximum load, varying the load between 1.0 and 10 mN. Three random measurements for each specimen were taken and mean values of nanohardness were calculated directly by the software connected to the nanoindenter. Once the hardness values of the specimen were determined, the elastic modulus was obtained mathematically from the load-displacement curve. There was no separate test required for determination of elastic modulus.

Evaluation of color stability

Preparation of staining solution

In the present study, a coffee solution was used as a staining solution[14,15] due to the fact that coffee was the most chromogenic substance in comparison with other staining substances, such as tea and cola drinks. The coffee solution was prepared by adding 250 mL of boiled distilled water to 10 g of coffee powder (Nescafe, Nestle Brazil, Brazil) in a coffee cup and stirred until it cooled. The coffee was filtered using filter net to remove any residues and stored in an airtight amber colored bottle. The coffee solution was prepared freshly every week till the conclusion of the staining process. In addition, the stored coffee solution was stirred daily for 1 min to reduce the precipitation of the particles.

Specimen preparation and testing

Ten samples (20 mm length) of each brand of coated archwires were prepared for calorimetric measurement. The wires (n = 10) were approximated to each other (touching each other by their sides), and their ends were joined by an adhesive resin (Transbond XT, 3M ESPE, St Paul, MN, USA) and the coating facing in the same direction. The reason for such a sample preparation is because it is impossible to measure the color measurement of a thin width of archwires. The wires should have at least 7 mm of width for proper color measurement.[15] The samples were stored in distilled water for 24 h at room temperature and after 24 h the initial color measurement (T_0) was performed.

The color measurement was made using Commission Internationale de l'Eclairege L*a*b* (CIELAB) color space using color eye 7000 spectrophotometer (GretagMacbeth, New Windsor, NY, USA) [Figure 3]. The CIE L*a*b* color system is a quantitative systems with rectangular coordinates that allow an objective color measurement which measures the value and chroma of the sample on L*a*b* coordinates: L* measures the lightness of the color from black (L* = 0) to white (L* = 100) (a value of 100 corresponds to perfect white and that of zero to black); a* – color in the red (a* > 0) and green (a* < 0) dimension; and b* – color in the yellow (b* > 0) and blue (b* < 0) dimension. The total color differences (ΔEab*) were calculated based on the following formula $\Delta Elab^* = ([\Delta L^*]^2 + [\Delta a^*]^2 + [\Delta b^*]^2)^{1/2}$.

The values obtained by L*a*b* color space were converted to the National Bureau of Standards (NBS) units to relate the changes to a clinical environment. The NBS interpretation included NBS units 0.0–0.5: Trace-extremely slight change, 0.5–1.5: Slight - slight change, 1.5–3.0: Noticeable-perceivable, 3.0–6.0: Appreciable-marked change, 6.0–12.0: Much-extremely marked change, 12.0 or more: Very much-change to other color.

After the first measurement (T0), the wire samples were placed in a container filled with the prepared staining coffee solution. Color measurements were repeated after 7 days (T_1), 14 days (T_2), 21 days (T_3), and 28 days (T_4) of immersion in the staining solution. For every color measurement, the sample was taken out from the container rinsed with distilled water for 5 min then dried using a tissue paper.

Statistical analysis

The experimental results were analyzed using Statistical Package for the Social Science (SPSS) software (version 18.0, SPSS Inc., Chicago, Illinois, USA). A descriptive statistic of mean and standard deviations (SD) was calculated for surface roughness, hardness, modulus of elasticity and color change values. While analyses of variance and Tukey's *post hoc* test were applied for comparing the differences among these means. The statistical significance level was determined at $P = 0.05$.

Results

Surface roughness

The mean (±SD) surface roughness values are shown in Table 1. For the surface roughness parameter in the experimental group, epoxy (1.517 ± 0.071) had the highest value and rhodium (0.297 ± 0.015) the lowest value. The mean (±SD) surface roughness for control group was 0.293 (±0.007). The surface roughness measurements showed statistically significant differences ($P < 0.05$). Intergroup comparisons revealed that there were differences between groups. The surface roughness was significantly different from each other for all intergroup comparisons ($P < 0.05$) except between rhodium and control groups ($P > 0.05$) [Table 2].

Nano-mechanical (nanohardness and elastic modulus) properties test groups

The mean (±SD) nanohardness values are shown in Table 1. Among the experimental group, rhodium (0.186 ± 0.036) had the highest value and epoxy (0.143 ± 0.100) had the lowest value of nanohardness. The mean (±SD) hardness for control group was 3.249 (±0.384). Nanohardness measurements showed statistically significant differences ($P < 0.05$). However, intergroup comparisons revealed statistically significant differences only between control and the experimental groups ($P < 0.05$) [Table 2].

Table 1: Mean±standard deviation values of surface roughness, nanohardness and elastic modulus of different group of archwires (*P<0.05)

Properties	Arch wires	Mean±SD	Minimum	Maximum	P
Surface roughness	Control	0.293±0.007	0.283	0.302	0.000*
	Epoxy	1.517±0.071	1.421	1.586	
	Rhodium	0.297±0.015	0.274	0.312	
	Teflon	0.857±0.014	0.841	0.875	
Nano-hardness	Control	3.249±0.384	2.870	3.770	0.000*
	Epoxy	0.143±0.100	0.018	0.228	
	Rhodium	0.186±0.037	0.121	0.207	
	Teflon	0.168±0.032	0.126	0.196	
Elastic modulus	Control	56.413±4.593	51.315	60.854	0.000*
	Epoxy	4.409±2.109	2.165	7.587	
	Rhodium	5.063±0.448	4.293	5.435	
	Teflon	5.345±0.508	4.739	5.965	

*P<0.05. SD=Standard deviation

Similarly, the mean (±SD) elastic modulus values are shown in Table 1. In the experimental group, Teflon (5.345 ± 0.508) had the highest value and epoxy (4.409 ± 2.109) had the lowest value. The mean (±SD) elastic modulus for control group was 56.413 (±4.593). Elastic modulus measurements showed statistically significant differences ($P < 0.05$). Intergroup comparisons revealed that there were statistically significant differences between control and the experimental groups ($P < 0.05$) [Table 2].

The color differences reported using the ΔE* and NBS units for the NiTi-coated esthetic archwires after each immersion period are presented in Figures 4 and 5. The ΔE* values at 1, 2, 3, and 4 weeks after immersion for Epoxy, rhodium, and Teflon ranged from 0.016 to 0.036, 0.024 to 0.039, and from 0.036 to 0.085, respectively. There were a statistically significant differences in ΔE* among the measured values of the three experimental groups ($P < 0.05$). The ΔE* for epoxy, rhodium, and Teflon control group was 0.013, 0.018, and 0.021, respectively [Figure 4].

The NBS units at 1, 2, 3, and 4 weeks after immersion for Epoxy, rhodium, and Teflon ranged from 0.014 to 0.033, 0.022 to 0.036, and from 0.033 to 0.078, respectively. The results showed that all of the samples exhibited color changes according to the

Table 2: Comparison of mean differences of surface roughness, nanohardness and elastic modulus of different group of archwires

Properties	Archwires groups		Mean difference	P	95% CI	
					Lower bound	Upper bound
Surface roughness	Rhodium	Control	0.004	0.998	−0.069	0.078
	Epoxy	Control	1.224	0.000*	1.151	1.298
	Teflon	Control	0.564	0.000*	0.490	0.637
	Epoxy	Rhodium	1.220	0.000*	1.147	1.293
	Epoxy	Teflon	0.660	0.000*	0.587	0.734
	Teflon	Rhodium	0.560	0.000*	0.486	0.633
Nano-hardness	Control	Epoxy	3.096	0.000*	2.714	3.477
	Control	Rhodium	3.053	0.000*	2.671	3.434
	Control	Teflon	3.072	0.000*	2.690	3.453
	Rhodium	Epoxy	0.043	0.986	−0.318	0.403
	Rhodium	Teflon	0.019	0.999	−0.341	0.379
	Teflon	Epoxy	0.024	0.997	−0.336	0.384
Elastic modulus	Control	Epoxy	52.004	0.000*	47.051	56.957
	Control	Rhodium	51.350	0.000*	46.396	56.303
	Control	Teflon	51.067	0.000*	46.114	56.021
	Rhodium	Epoxy	0.654	0.978	−4.016	5.324
	Teflon	Epoxy	0.937	0.939	−3.734	5.607
	Teflon	Rhodium	0.283	0.998	−4.388	4.953

*P<0.05. CI=Confidence interval, SE=Standard error

NBS values, which ranged from 0.014 to 0.078. The color change values after immersion for 1, 2, 3, and 4 weeks were <0.5 for all three NiTi-coated esthetic archwires, and only "trace" (extremely slight change) color changes were observed according to the NBS units. Furthermore, there were statistically significant differences in the color-change values for the NiTi-coated esthetic archwires in the immersion periods ($P < 0.05$). The NBS units for Epoxy, rhodium, and Teflon control group was 0.012, 0.016, and 0.019, respectively [Figure 5].

ΔE* values were converted to NBS units by the equation: NBS units = ΔE* × 0.92. For each NiTi-coated esthetic archwires, there were a statistically significant differences in color change for the immersion periods ($P < 0.05$), as shown in Table 3.

Discussion

The present study evaluated the surface roughness and nanomechanical properties of three esthetic-coated archwires and uncoated counterparts from the same company. In addition, the color stability of esthetic archwires was assessed after immersion in staining solution at an interval of 7, 14, 21, and 28 days.

The growing esthetic concerns of orthodontic patients has led to the development of various esthetic orthodontic products such as ceramic brackets,[16] lingual orthodontics,[17] and Invisalign®.[18] The tooth-colored archwires created much hype in orthodontics and are in growing demand in modern orthodontic practice. Since then many researchers have studied the esthetic archwires evaluating the clinical performance *in vivo* and *in vitro*.[2,10,19-22]

The roughness of a material is a measure of texture of a surface, and it influences how an object will react with its environment.[23] The surface roughness of orthodontic wires is an important factor in deciding the effectiveness of archwire-guided tooth movement. The surface quality of wires affects the area of surface contact and influences the corrosion behavior and biocompatibility of the archwires, increase caries and gingivitis risk in addition to modifying the esthetics and efficiency of the orthodontic components. It has been reported that archwire surface structure is

Figure 1: The noncontact surface profilometer – GTR1 - Bruker Campbell CA95008, USA

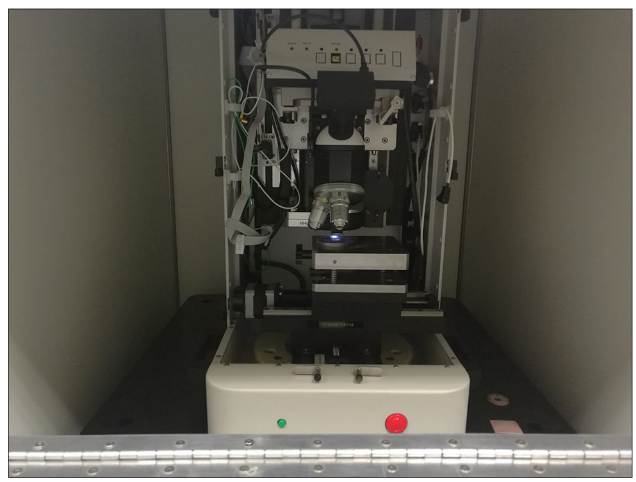

Figure 2: Nano indenter Bruker, Tucson, AZ, USA

Figure 3: Color Eye 7000 spectrophotometer (Gretag Macbetch, New Windsor, NY, USA)

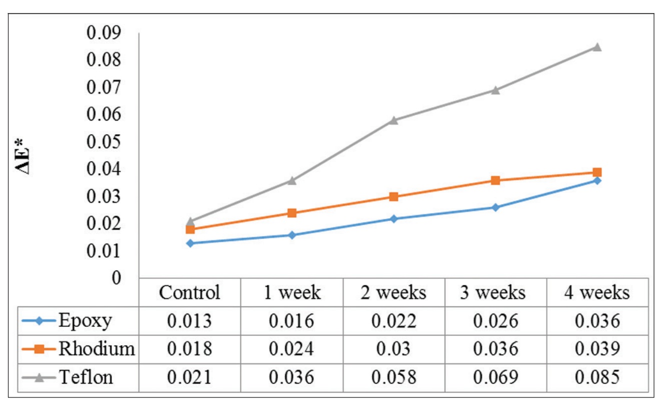

Figure 4: Color differences measured in ΔE* after immersion in the coffee solution

Table 3: Color differences measured in ΔE* and National Bureau of Standards units after immersion in the coffee solution

Wires	Control		1 week		2 weeks		3 weeks		4 weeks	
	ΔE*	NBS units	ΔE*	NBS units	ΔE*	NBS units	ΔE*	NBS units	ΔE*	NBS units
Epoxy	0.013	0.012	0.016	0.014	0.022	0.020	0.026	0.024	0.036	0.033
Rhodium	0.018	0.016	0.024	0.022	0.030	0.028	0.036	0.033	0.039	0.036
Teflon	0.021	0.019	0.036	0.033	0.058	0.053	0.069	0.063	0.085	0.078

**P*<0.05 Significant value. NBS=National Bureau of Standards

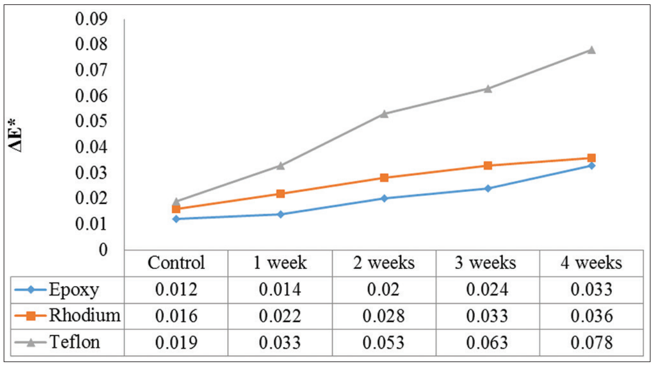

Figure 5: Color differences measured in National Bureau of Standards units after immersion in the coffee solution

influenced by material coating, manufacturing process, and manufacturer.[24,25]

The present study evaluated surface roughness of the archwires using a noncontact profilometer as it is an ideal and primary technique to investigate the surface roughness of the archwires.[23] However, few researchers criticized the use of profilometry to measure surface roughness due to its inability to measure the entire surface area.[25] As the wires were scanned along a preselected area using contact scanning white light interferometry.

In this study, the surface roughness measurement of the esthetic archwires demonstrated high values with epoxy-coated archwires and the least for uncoated archwires and rhodium-coated archwires. Furthermore, there was a significant difference in roughness values observed among the coated archwires. This difference could be due to the different coating present on the archwires. This finding is in agreement with the outcome of the previously reported studies.[23,25] The significant difference among the tested archwires in this study suggestive of careful consideration to ensure proper selection of the esthetic archwires so as to minimize the adverse effects caused by high surface roughness. This study highlighted rhodium-coated archwires as the best-coated archwires.

The nanomechanical properties (nanohardness and elastic modulus) of the coated archwires were obtained with application of low load to the coating present on the wires rather than the wire material itself.[1] The nanohardness of the coated archwires in the present study was in the range of 0.14–0.18 Gpa and elastic modulus ranged between 4.40 and 5.34 Gpa. There was no significant difference among the esthetic archwires for both the nanohardness and elastic modulus measurements. This finding is in agreement with the previous work by da Silva *et al*. where they evaluated four coated esthetic archwires and found similar values of hardness and elastic modulus.[1] This finding is suggestive of the fact that the different coatings on the archwires did not influence the mechanical properties significantly. This view was supported by a study of Albuquerque *et al*. in which two esthetic archwires were evaluated and concluded that mechanical properties and surface morphology is significantly altered by the coating process and not the type of coating.[20]

The color stability of coated esthetic archwires plays a crucial role during orthodontic treatment. Previous color measurements studies have focused on the color stability of esthetic orthodontic appliances such as brackets and ligatures and literature regarding the color stability of archwires are scarce.[15]

The color of coated esthetic archwires should compete equally with the color of the esthetic brackets, natural teeth, and other orthodontic components. However, the colors of natural teeth differ according to race, gender, age, and visual perception of the observer. To overcome problems of the visual color comparison, instrumental measurements such as spectrophotometer are used to assess the color stability of the coated esthetic archwires. The CIE L*a*b* color space is the commonly used and widely accepted color measurement system as it is very ideal determination of small color differences.[14]

The present study used NBS rating system using DE* values in relating the color stability of the orthodontic archwires. The reason behind using NBS system is that it counters the differences and disagreements in evaluating the "perceptibility" of color differences which was differently adopted by different authors or researchers. The NBS rating system offers absolute criteria by which DE* values can be converted to definitions with clinical significance.[14]

In this study, the evaluation of color stability of the archwires demonstrated trace to slight (0.0–0.5 NBS units) color changes after 4 weeks of immersion in staining solution according to the NBS interpretation. Teflon-coated archwires showed the highest change in color measurements following 4 weeks of immersion (0.5–1.5 NBS units/slight change). Epoxy and Rhodium showed trace (0.0–0.5 NBS units/slight change) color changes among all the samples. The color change value between rhodium and epoxy did not show any significant difference.

In a study conducted by da Silva *et al*., esthetic archwires demonstrated noticeable or perceivable color change after 3 weeks of immersion.[14] The present study finding is contrary to the outcomes of previously reported studies by da Silva *et al*. and Mujawar *et al*., in which aesthetic archwires showed noticeable color change after 21 days of immersion in staining solution.[14,26] On the other hand, Inami *et al*. evaluated the color stability of esthetic archwires and reported the slight change in color which is in line with the findings of the present study.[15]

The main limitations of the present study were that we could not able to compare the results with other studies due to the lack of similar studies.

Conclusion

The present study concluded that the rhodium-coated archwires demonstrated surface roughness almost similar to that of uncoated archwires. However, Teflon and epoxy coated archwires exhibited a significant difference in surface roughness compared to uncoated archwires. Uncoated archwires presented with high hardness values compared to coated archwires. Teflon-coated archwires demonstrated significant changes in color values after 4 weeks of immersion in staining solution, yet the color changes were a slight change according to NBS definition. Further studies are required to compare other mechanical properties and fluorescence of the wires.

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Nil.

Conflicts of interest

There are no conflicts of interest.

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