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Original Article

# Evaluation of the surface properties of modified Ni-Ti arch wires

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## Abstract

**Aim:** Materials used in orthodontic treatment can cause corrosion and metal ion release to the mouth. This study was aimed to examine the changes in the friction coefficients and corrosion resistance of the modified Ni-Ti arch wires.

**Methodology:** Our sample consisted of 16 Ni-Ti arch wires (0.016 × 0.022 inches). Coating bathes involving Ni-Ti, Ni-Ti-Mo, and Ni-Ti-Cr were prepared for arch wires coating. New surfaces were analyzed by Scanning Electron Microscope (SEM), and the crystallographic structure was determined by X-rays. The friction coefficient of the coated wires was determined by nanoindentation. Artificial saliva was prepared as a corrosive condition. Impedance measurements were performed with an electrochemical analyzer. The AC impedance spectroscopy measurements were performed after 4 hours, 7, 15, 30, 45, and 60 days.

**Results:** All groups were more resistant than the control group (-447 mV) [group 1 (-388 mV), group 2 (-328 mV), group 3 (-327 mV), group 4 (-427 mV), group 5 (-395 mV), group 6 (-366 mV), and group 7 (-380 mV)]. Friction coefficients of group 5 (0.252) and group 6 (0.265) were observed to be lower than those of the control group (0.288).

**Conclusion:** In the results of our study, it was shown that the surface properties of Ni-Ti wires could be electrochemically improved. The coated wires (group 5 and group 6) had a remarkable corrosion resistance and reduced friction. The protocols that we used should help provide better-quality surfaces.

**Keywords:** arch wire, coating, corrosion, friction, Ni-Ti

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## Introduction

Malocclusion treatment in a 3-dimensional manner may be provided by orthodontic treatment with fixed appliances. Orthodontic wires producing biomechanical forces via brackets for tooth movement are pivotal in orthodontic practice (1). High levels of effectiveness and compliance regarding the timing and outcomes of treatment, when compared to treatments with removable appliances, are achieved with fixed orthodontic treatment (2).

The effectiveness of orthodontic tooth movement is affected by both biological and mechanical variables. Friction is one of the most intensively studied issues due to its complexity and role in tooth movement. The orthodontic tooth movement occurs when an orthodontic force applied to the tooth exceeds static friction (3). The amount of friction is affected by several physical variables, including bracket type and wire material (4), type of wire application (5), surface characteristics (6), shape and size (3), ligature type (7), and method of wire attachment to bracket (8). It is impossible to eliminate friction in sliding mechanics

completely. Thus, orthodontists should be informed about the factors influencing friction and how to control these factors in individual phases of treatment when needed (3).

In recent years, the use of self-ligating brackets (3), brackets with modified slot surfaces and geometry (3, 9, 10), light-ligating steel wires (8), elastic ligatures with novel designs (11), and to coat the external surfaces of orthodontic wires (12) have been recommended.

In the oral cavity, electrolytic and electrochemical corrosion occurs due to humidified media (13). Corrosion is an ongoing decomposition process due to comprehensive alterations in temperature, pH, microbial features, and saliva-related factors (14). If the metal is not stable, it may undergo attrition, releasing metal ions into the oral fluid. This process is maintained until the chemical potential is achieved or the ion flow is hampered. For example, a passive film formation that produces a barrier to abolish contact between metal and fluid may decelerate the ion formation rate (15). In this study, we hypothesize a reduction in friction coefficients and improvement in resistance against corrosion by modifying the surface characteristics of Ni-Ti arch wires used in orthodontic treatment.

## Materials and Methods

The study was conducted on eight groups, including the control, using 16 Ni-Ti arch wires (0.016 × 0.22 inches; American Orthodontics, USA). The Ni-Ti arch wires were purchased and coated with a UV-insulator dye, with the surface area being constant. Then, electrodes were washed with acetone: ethanol mixture (1:1) and distilled water. We prepared Ni-Ti-Mo baths containing molybdate salt at three different concentrations and Ni-Ti-Cr baths containing chrome salt at three concentrations to coat the Ni-Ti arch wires. The coating was performed at 0.94 mA over 293 seconds by a single-cell three-electrode system using chronopotentiometry. A platinum plate was used as a counter electrode (surface area: 0.50 $cm^2$), whereas Ag/AgCl (3M, KCl) was used as a reference electrode. During the coating baths for the Ni-Ti alloy (group 1), 1.29 $g/dm^3$ $NiSO_4$, 0.27 $g/dm^3$ $NiCl_2$, 0.12 $g/dm^3$ $Na_3C_6H_5O_7$, 0.48 $g/dm^3$ $H_3BO_3$, and 0.025 $g/dm^3$ $TiO_2$ (nanoparticle) were used in a standard manner (16). In addition, 0.0015 (group 2), 0.0025 (group 3), and 0.0035 (group 4) $g/dm^3$ $CrCl_3$ were added to Ni-Ti-Cr baths, whereas in the other groups, 0.0015 (group 5), 0.0025 (group 6), and 0.0035 (group 7) $g/dm^3$ $Na_2MoO_4$ were added to Ni-Ti-Mo baths, respectively.

Artificial saliva as a corrosive media was prepared to place coated wires. The artificial saliva contained 0.20 $g/dm^3$ $K_2HPO_4$, 1.20 $g/dm^3$ KCl, 0.33 $g/dm^3$ KSCN, 0.26 $g/dm^3$ $Na_2HPO_4$, 0.70 $g/dm^3$ NaCl, and 1.50 $g/dm^3$ $NaHCO_3$. Additionally, 1.50 $g/dm^3$ urea and lactic acid were added to the solution until a pH value of 6.7 was achieved (17).

Surface images were assessed in the control group and newly formed surfaces with SEM (JEOL 5500/OXFORD Inca-X). Additionally, the crystallographic structure of the arch wires from each group was determined using an X-ray (RIGAKU Smartlab, Japan).

The coated wires were placed into artificial saliva, used as the corrosive media at 37.5°C. The AC impedance spectroscopy measurements were performed in open-circuit potential on the fourth hour and on the seventh, $15^{th}$, $30^{th}$, $45^{th}$, and $60^{th}$ experimental days. The measurements were obtained using a $10^5$–$10^{-3}$ frequency and an amplitude of 7 mV at 37.5°C, as shown in the Nyquist diagram.

On the 60th day after the wires were placed into the corrosive media, they were scanned, beginning from equilibrium potential ($E_{cor}$) to cathodic and anodic directions up to + 1800 mV at a scan rate of 4 mV/seconds by the electrochemical analyzer (CH Instruments, 608 C, Austin, USA). Corrosion performance was assessed using AC impedance spectroscopy, Tafel curves, and open-circuit potential-time graphics.

The friction coefficients of the coated wires were compared with a Nano Intender analyzer (HYSITRON TI 950 TriboIndenter, Hysitron Inc., USA). The coated wires in the control and alloy groups were cut into pieces (1 cm in size). Friction coefficients were measured by creating scratches (300 nm at depth) on samples over 15 s.

## Statistical analysis

The data were analyzed using SPSS software version 21.0 (IBM Corp., Armonk, New York, USA). The distribution of friction coefficients was assessed with a Shapiro-Wilk test, in which a skewed distribution was indicated. Thus, data regarding friction coefficients were evaluated with non-parametric Kruskal-Wallis and Mann-Whitney U tests. A p-value < 0.05 was considered statistically significant.

## Results

### Crystallographic structure assessments

When the control group was assessed, it was seen that there were highly sharp peaks at 42.31, 77.44, and 61.45 degrees in the range of 2 theta. These peak values were obtained from ‘‘The International Centre for Diffraction Data’’ (ICDD) 01-071-7598 database.

Contrary to peaks in the control group, coated Ni-Ti crystals were observed at peaks of 44, 51, and 76 degrees in group 1, 44 and 50 degrees in group 2, 41 and 51 degrees in group 3 and group 4. Peaks of 43, 51, and 75 degrees were observed in group 6, and 61.5 and 77.3 degrees in group 7. With such peaks, it was shown that the Ni-Ti-Cr-Mo crystals accumulated on the Ni-Ti wires.

### Nano mechanical assessment

Based on statistical results, there was a significant difference between friction coefficients (Table 1 and

Fig. 1 and 2; $p < 0.05$). When coatings were assessed in all concentrations, the friction coefficient was found to increase in groups 1 (0.309), 2 (0.530), 3 (0.314), and 4 (0.395) compared to the control group (0.288).

In the Ni-Ti-Mo coated wires, the friction coefficient decreased in groups 5 (0.252)

## SEM Images

SEM images of the coated arch wires in the different groups (Fig. 3).

**Table 1.** Statistical results of the friction coefficients for the different groups ($p < 0.05$).

| | Control | Group 1 | Group 2 | Group 3 | Group 4 | Group 5 | Group 6 | Group 7 |
|---|---|---|---|---|---|---|---|---|
| Mean | ,290 | ,308 | ,515 | ,325 | ,420 | ,252 | ,257 | ,395 |
| Median | ,288 | ,309 | ,530 | ,314 | ,395 | ,252 | ,265 | ,429 |
| Std. Deviation | ,010 | ,004 | ,096 | ,049 | ,109 | ,039 | ,045 | ,089 |
| Minimum | ,266 | ,299 | ,269 | ,231 | ,093 | ,146 | ,123 | ,160 |
| Maximum | ,314 | ,319 | ,719 | ,485 | ,688 | ,321 | ,329 | ,526 |

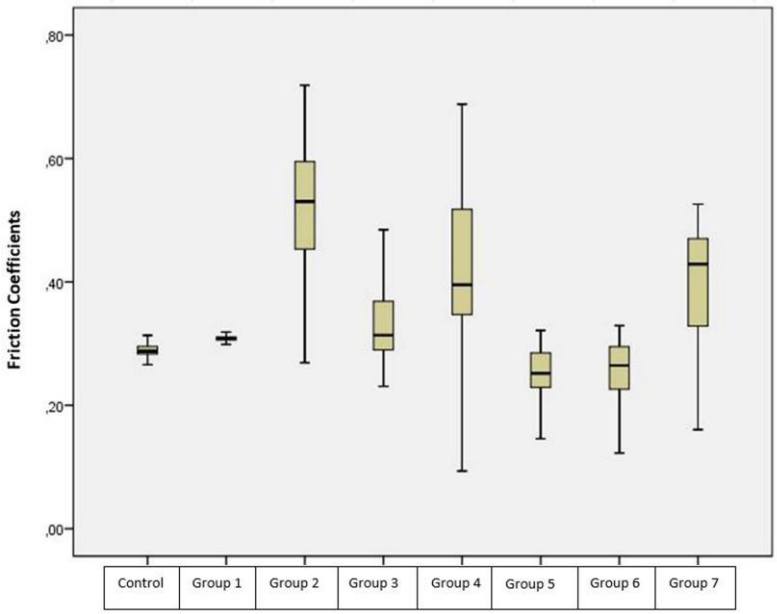



**Figure 1.** Box plot graphs of the friction coefficients

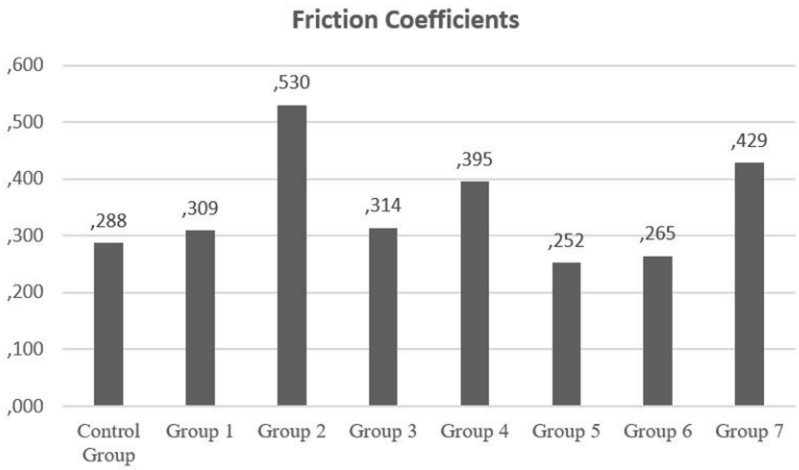



**Figure 2.** Column graphs after nanoindentation

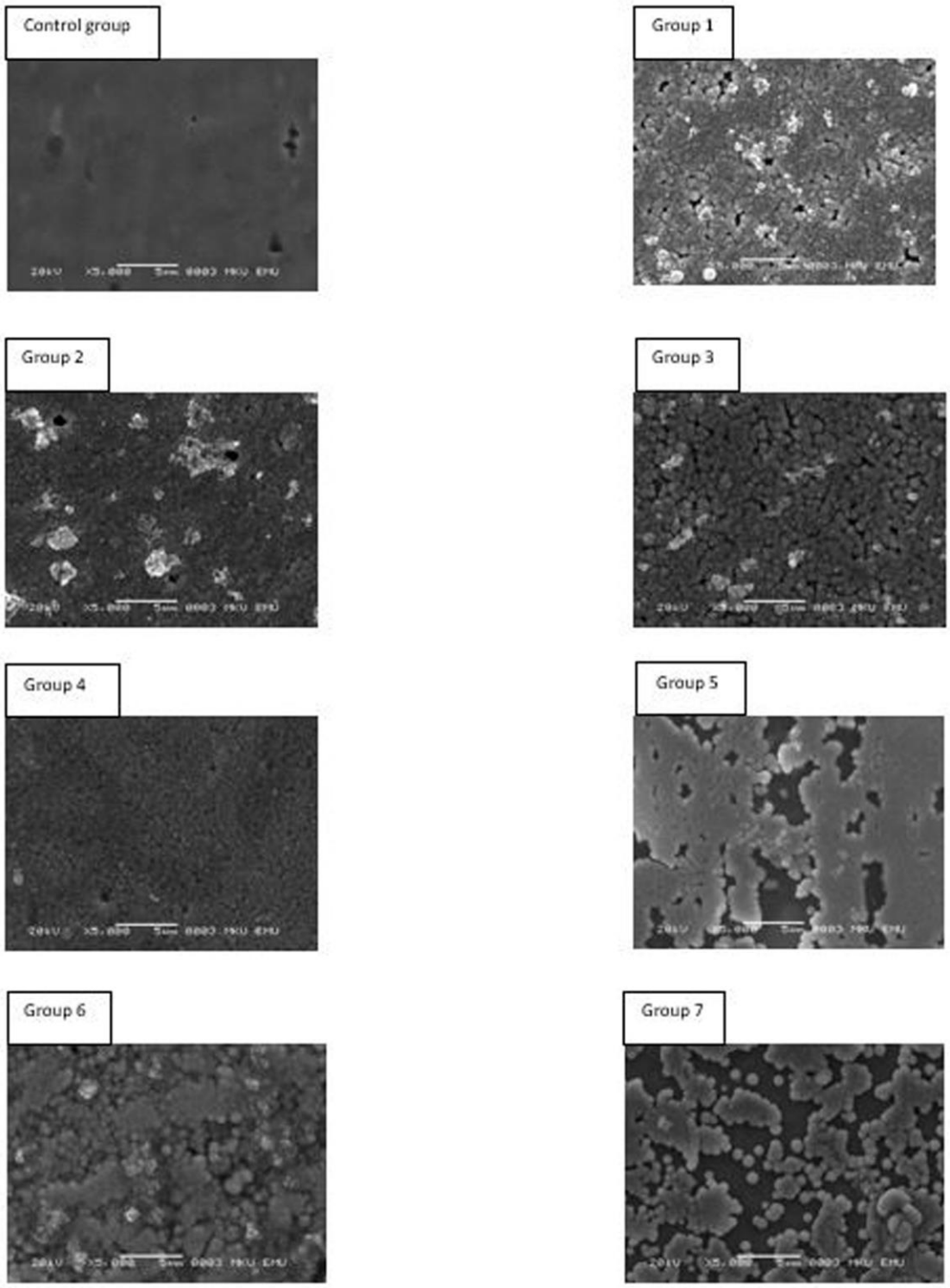



Figure 3. SEM images at 5000x magnification.

## Tafel Curves

In Figure 4, the corrosion potential values ($E_{cor}$) of electrodes in groups 1, 2, 3, and 4 were in a positive zone compared to controls. It was observed that current values were lower in the control group when compared to the coated electrodes.

In Figure 5, it was observed that the corrosion potential value ($E_{cor}$) of electrodes in group 7 was in the negative zone, whereas it was in a positive zone in group 6 compared to controls. It was observed that current values were lower in the control group compared to coated electrodes. A more positive $E_{cor}$ value in group 6 is a suggestion of fewer active areas over the electrode surface. The finding that the same electrode had the lowest current value is a suggestion that the superficial coating has barrier characteristics and resistance to corrosive media.

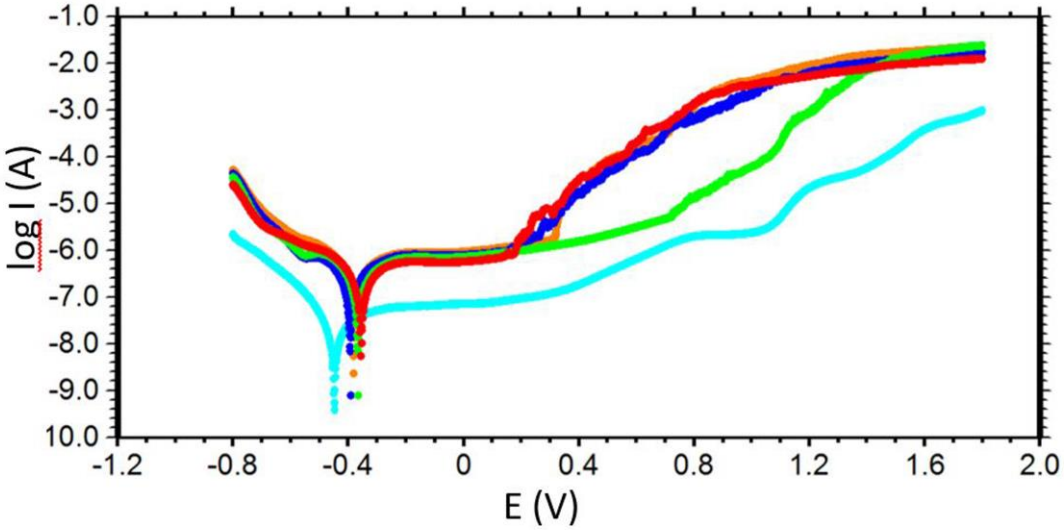



Figure 4. Tafel curves of the control group (blue), groups 1 (orange), 2 (navy blue), 3 (green), 4 (red) obtained on the 60th day after placement in artificial saliva.

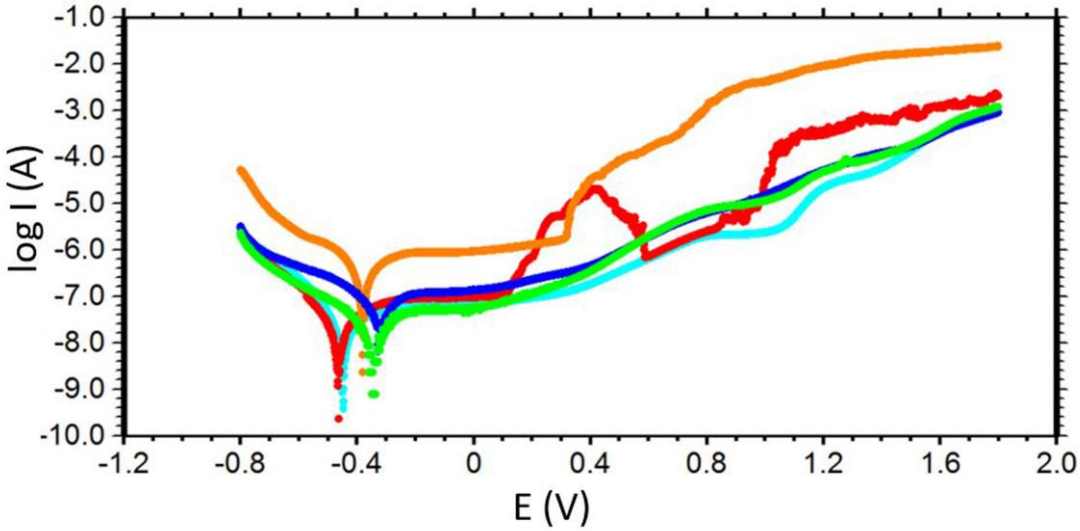



Figure 5. Tafel curves of the control group (blue), groups 1 (orange), 5 (navy blue), 6 (green), 7 (red) obtained on the 60th after placement in artificial saliva

## Impedance Measurement

In the control group, resistance against a stable oxidation layer over the electrode surface and resistance corresponding to the metal-solution interface of pores in the coating was observed at zones corresponding to high- and low-frequency areas in the Nyquist diagram (Figure 6). It was observed that the resistance was highest at the fourth hour, with a gradual decrease until the 45th day. Again, after the 45th day, the resistance increased.

A single semicircle extending from a high-frequency zone to a low-frequency zone in the Nyquist diagram obtained at the fourth hour of the experiment was observed in group 1. The diameter of the semicircle is a representation of polarization resistance ($R_p$).

A single semicircle extending from a high-frequency zone to a low-frequency zone in the Nyquist diagram obtained at the fourth hour of the experiment

was observed in group 2. However, a linear component appeared over time.

The magnitude of resistance corresponding to $R_p$ in the fourth experimental hour was more significant in group 3 than in group 2.

The $R_p$ value at the fourth experimental hour was smaller in group 4 than in group 3. The $R_p$ value at the fourth hour was markedly lower. However, increased resistance was observed on the 60$^{th}$ day. The $R_p$ value at the fourth hour was lower in group 6 than in group 5. The slope of the linear component in the low-frequency zone increased over time.

The $R_p$ value at the fourth hour was greater in group 7 than in groups 5 and 6.

In Figures 7 and 8, the Nyquist diagrams obtained on the 60$^{th}$ day in groups 1, 2, 3, 4, 5, 6, and 7, aimed to determine coating durability against prolonged immersion periods, are shown.

Group 6 had the highest resistance against corrosion on the 60$^{th}$ experimental day, as indicated by the greatest linear component (Fig. 8).

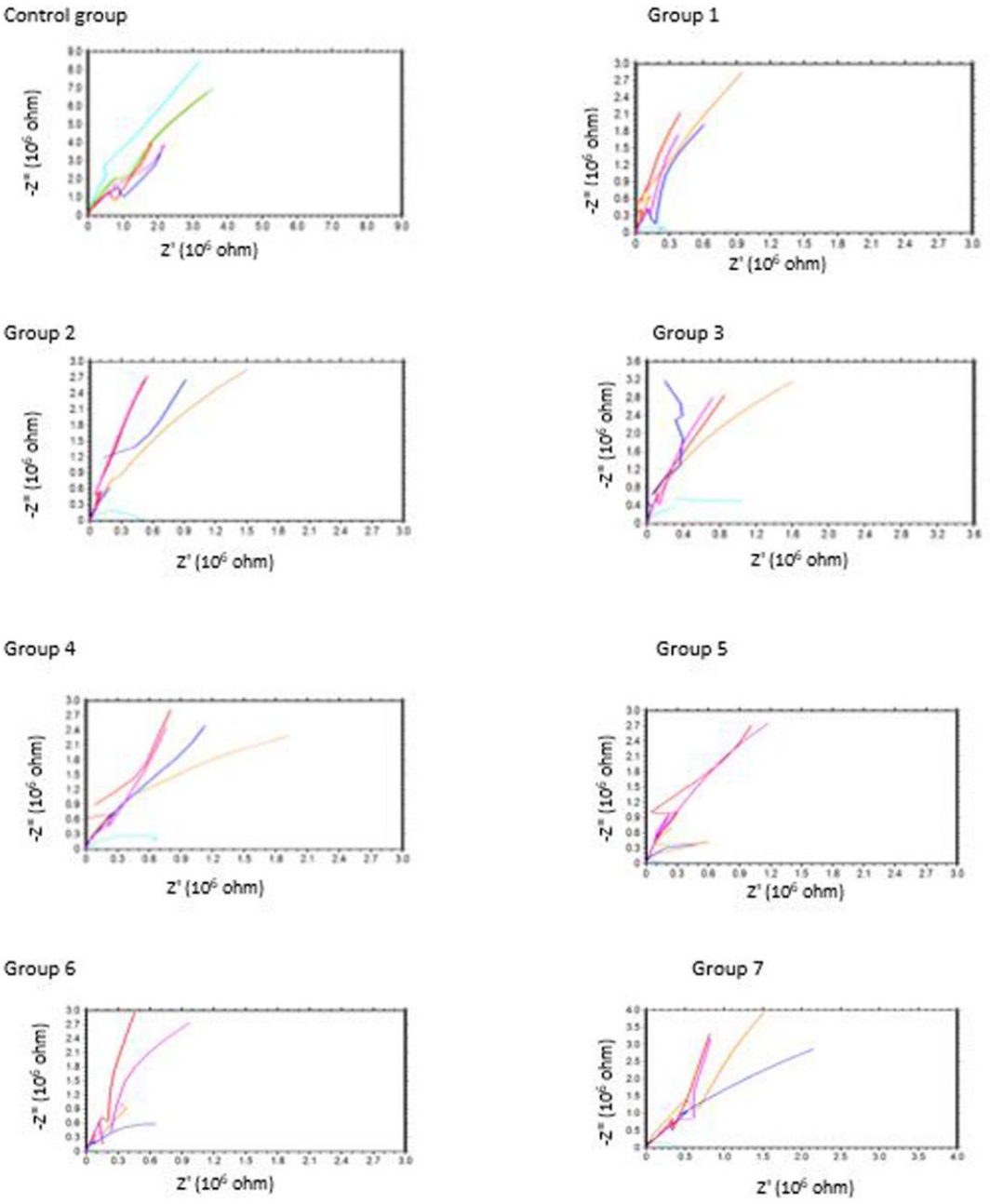



**Figure 6.** The Nyquist diagrams obtained on the fourth (blue) and 24 hours (green) and at the seventh (orange), 30$^{th}$ (navy blue), 45$^{th}$ (purple) and 60$^{th}$ (red) days after the saliva placement.

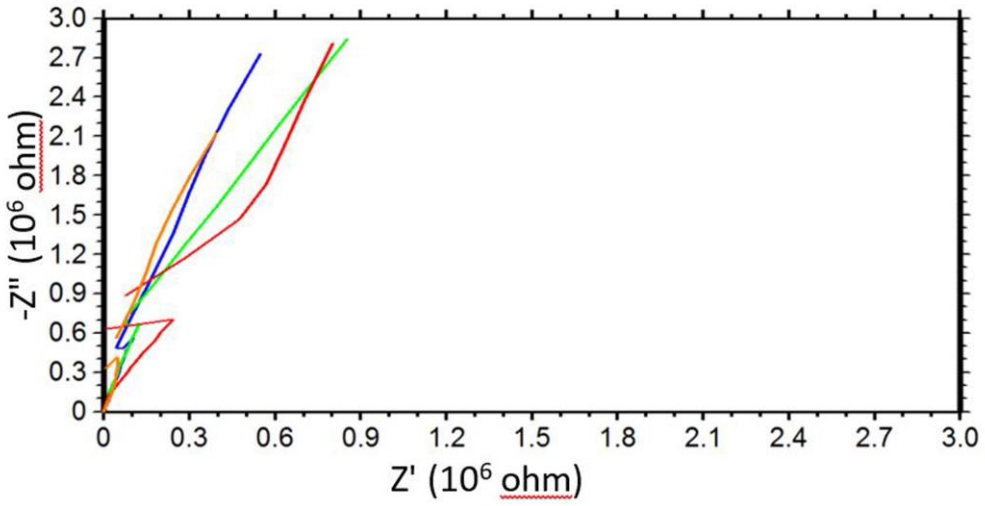



**Figure 7.** The Nyquist diagrams obtained on day 60th in groups 1 (orange), 2 (navy blue), 3 (green) and 4 (red)

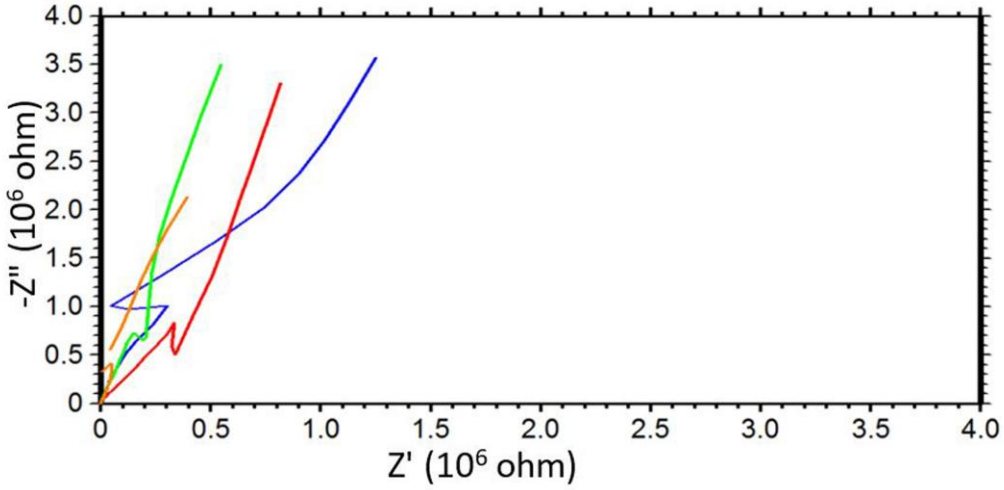



**Figure 8.** The Nyquist diagrams obtained on day 60th in groups 1 (orange), 5 (navy blue), 6 (green) and 7 (red)

## Discussion

Orthodontic arch wires should have numerous features, such as high durability, low stiffness, high elasticity, excellent frictional characteristics, and corrosion resistance, to be considered effective intraoral materials (18, 19).

The natural corrosion of the arch wires occurring in a prolonged period is mimicked in in vitro anodic polarization studies. At baseline, open-circuit potentials relative to an electrolyte were determined for samples in this method. Artificial saliva (20), normal saline (21), and Ringer's solution (22) are among the commonly used electrolytes in corrosion studies. The initial point of the anodic polarization scanning was guided by open-circuit potentials. By this method, the minimum potential required to initiate corrosion is determined for the given material (23). The wires showing degradation at minimum potential had the highest corrosion predisposition.

In a study by Kim and Johnson (24), corrosion resistance was assessed for epoxy, nitrite, and conventional wires. In this study, the degradation potential was higher for epoxy (1800 mV), but no difference was detected between nitrite and conventional Ni-Ti wires (300 mV in each). In another study by Krishan et al. (25), both nitrite ion- and resin-modified wires (333 and 346 mV, respectively) had higher degradation potentials than conventional wires (204 mV). This finding may be attributed to the different manufacturers of the wires used in the study. The corrosion resistance of nitrite ion-modified wires is in agreement with the findings of Tan et al. (26). However, black oxide-coated wires had the highest anticorrosive features (526 mV), as observed by Krishan et al. (25). Similarly to the study by Krishan et al., all wires with surface modification showed higher anticorrosive features than conventional wires in our research.

Currently, there is an increasing tendency to use aesthetic materials such as Teflon, epoxy, or resin to modify the wire surfaces. Thus, corrosion performance merits assessment in these materials. In a study, Teflon (1181 mV) and gold (872 mV) coatings, considered appropriate aesthetic coatings for Ni-Ti wires, had maximum corrosion resistance (25). Although Teflon has a rough appearance in superficial SEM and Atomic Force Microscope (AFM) images, it still has the highest degradation potential. On the other hand, resin-coated wires did not have the degradation potential of Teflon (1186 mV) and gold (872 mV), although they had better performance (345 mV) than the conventional Ni-Ti wires used in the control group (25). Similarly, Ni-Ti, Ni-Ti-Cr, and Ni-Ti-Mo wires coated at different concentrations had more corrosion resistance than the uncoated wires in the control group of our study. Here, we believe that the differences in the results could be due to differences in corrosive media and coating materials.

In orthodontic wires, surface roughness has been assessed regarding friction, corrosion, and aesthetic aspects (27). Although gold-coated wires had good resistance to corrosion (872 mV), they did not have smooth topography on SEM and AFM (23). Surface roughness was lower, but it did not have better degradation potentials in nitrite ion-coated wires. Similar disproportionality was observed between surface roughness and degradation potential values in Black Diamond modified Ni-Ti wires. This group containing barium, carbon, and oxygen had the lowest surface roughness (74.112 nm). However, they did not have a higher degradation potential. Similarly, no direct relationship could be established between the degradation potential value and surface roughness in Teflon and resin-modified wires (25). In our study, friction coefficients were increased in coated wires with improved corrosion resistance, except for the wires in groups 5 and 6. When wires in groups 5 and 6 were compared to controls, corrosion resistance increased with a decreased friction coefficient.

In 2014, Bravo et al. assessed friction and corrosion by coating Ni-Ti wires with polyamide (28). They observed that the static and dynamic friction coefficients were lower in polymer wires than in beta-Ti, cp-Ti, Ni-Ti, and Ni-Ti-Cu wires. Based on these results, polymer-coated wires were associated with lower friction than the remaining four wires (beta-Ti, cp-Ti, Ni-Ti, and Ni-Ti-Cu) for two different bracket types (Ti-6A1–4 V and 316 stainless steel brackets) in both a dynamic and static manner (28).

In the same study, the degradation potential was 470 mV in standard Ni-Ti wires, whereas it was 870 mV in polyamide-coated wires, according to corrosion tests. Improvements in polyamide-coated wires regarding both friction and corrosion were observed in the results (28). In agreement with the study mentioned above, positive results were recorded in groups 5 and 6 regarding both corrosion and friction compared to conventional wires in our research.

Zhang et al. coated stainless steel orthodontic wires with nanoparticle carbon films (29), and they obtained smoother and harder surfaces in carbon-coated wires than in the control group. The obtained surface had decreased kinetic friction. Zhang et al. used Tafel curves for corrosion assessment (29), observing a significant reduction amounting to corrosion in carbon-coated stainless-steel wires placed in artificial saliva (29). Similar to our study, corrosion was assessed using Tafel curves, as in the study mentioned above. However, in our research, the Ni-Ti wires were coated with Ni-Ti, Ni-Ti-Cr, and Ni-Ti-Mo, whereas Zhang et al. used carbon to coat stainless steel wires. In our study, increased corrosion resistance and decreased friction were detected in groups 5 and 6, whereas only increased resistance was detected in groups 1, 2, 3, 4, and 7.

In our study, the corrosion potential was -447 mV in the control group, whereas it was -388 mV in group 1. The coated wires in baths containing low concentrations of $Cr^{+3}$ and $Mo^{+6}$, corrosion potentials were -395 mV in group 2, -366 mV in group 3, -380 mV in group 4, -328 mV in group 5, -327 mV in group 6, and -427 mV in group 7. The materials used in our study are advantageous, as they improve protection against corrosion with no loss in the metallic characteristics of wires and the need for additional passivating processes. Based on the results we obtained, group 6 had the best protection performance against corrosion.

On the other hand, when the friction coefficients of the Nano Indenter were assessed, the coated wires in groups 5 and 6 had lower friction coefficients. At the same time, friction coefficients were higher in the remaining groups than in the control group.

When friction coefficients were assessed in the Ni-Ti-Mo-coated wires according to variations in Mo content, the friction increased with increasing amounts of $Mo^{+6}$ salt. This finding was supported by the SEM images.

## Conclusions

In this study, by assessing the surface characteristics of Ni-Ti arch wires after modification, the following results were observed:

1) Ni-Ti wires could be coated by Ni-Ti alloy in citrate-sulfate containing baths and by Ni-Ti-Cr and Ni-Ti-Mo alloys in baths added to three different concentrations of $Mo^{+6}$ and $Cr^{+3}$ salts via chronopotentiometry at a current density of 50 $Ma/cm^2$. The coating thickness was calculated as 5 µm;

2) When coatings were compared to uncoated wires, the corrosion resistance of the alloy coatings of the wires was improved. The wires in group 6 had the best corrosion performance with a lower friction coefficient than the uncoated Ni-Ti wires;

3) The lowest friction coefficient was detected in group 5.

Based on the results, we concluded that surface characteristics could be improved in Ni-Ti wires by electrochemical techniques based on the results. There was no loss in the mechanical and chemical features of the wires as a result of such enhancement. Instead, an improvement was provided.

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**Author Contributions:** Conception – H.Y.; Design – F.B.Z.; Supervision – A.T.Ö.; Materials – H.Y.; Data Collection and/or Processing –F.B.Z.; Analysis and/or Interpretation – A.T.Ö.; Literature Review – H.Y.; Writer – F.B.Z.; Critical Review – A.T.Ö.

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