Original Article

Influence of intraoral application of antiseptics and fluorides during orthodontic treatment on corrosion and mechanical characteristics of nickel-titanium alloy in orthodontic appliances

Tihana Zibar Belasic[a]; Biljana Pejova[b]; Helena Otmacic Curkovic[c]; Ervin Kamenar[d]; Bojana Cetenovic[e]; Stjepan Spalj[f]

ABSTRACT

Objectives: To explore whether the commercial agents recommended for controlling dental biofilm formation had a significant effect in vivo on mechanical and corrosion properties of nickel-titanium (NiTi) alloy.

Materials and Methods: NiTi archwires (dimensions 0.508×0.508 mm) were collected from 36 orthodontic patients aged 13–42 years after a 3-month intraoral exposure. Three experimental groups were formed: (1) subjects conducting regular oral hygiene, (2) subjects who used fluorides for intensive prophylaxis for the first month, and (3) subjects who used chlorhexidine in the same manner. Corrosion behavior, surface characteristics, stiffness, hardness, and friction were analyzed.

Results: Exposure to intraoral conditions significantly reduced the stiffness and hardness of the NiTi alloy ($P \leq .015$). Fluoride tended to reduce stiffness and hardness more than did saliva or antiseptic, but not significantly. Roughness and friction were not significantly influenced by oral exposure. Intraoral aging predominantly produced general corrosion independent of the adjuvant prophylactic agent, although localized corrosion may also have occurred.

Conclusions: Fluorides and the antiseptic chlorhexidine do not increase corrosion more than saliva itself, nor do they further modify the mechanical properties of the NiTi alloy. (*Angle Orthod.* 2021;91:528–537.)

KEY WORDS: Antiseptics; Corrosion; Fluorides; Nickel; Titanium

INTRODUCTION

Every orthodontic treatment increases the risk of damaging the tooth surface and the surrounding soft tissues as a result of the accumulation of dental biofilm.[1,2] The greatest prophylaxis challenge in orthodontics is the control of dental biofilm to consequently avoid the occurrence of white spot lesions and gingival inflammation. Practicing adequate oral hygiene is the general recommendation for avoiding such side effects. Additionally, suggested preventive methods

[a] Research Assistant, Department of Orthodontics, University of Rijeka, Faculty of Dental Medicine, Rijeka, Croatia.
[b] Professor, Institute of Chemistry, Ss. Cyril and Methodius University of Skopje, Faculty of Science, Skopje, North Macedonia.
[c] Associate Professor, Department of Electrochemistry, University of Zagreb, Faculty of Chemical Engineering and Technology, Zagreb, Croatia.
[d] Assistant Professor, Department of Mechanical Engineering Design, University of Rijeka, Faculty of Engineering; and Center for Micro- and Nanosciences and Technologies, University of Rijeka, Rijeka, Croatia.
[e] Research Associate, Department of Physical Chemistry, Vinca Institute of Nuclear Sciences, University of Belgrade, Belgrade, Serbia.
[f] Professor, Department of Orthodontics, University of Rijeka, Faculty of Dental Medicine, Rijeka; and Department of Dental Medicine 1, J. J. Strossmayer University of Osijek, Faculty of Dental Medicine and Health, Osijek, Croatia.
Corresponding author: Tihana Zibar Belasic, Assistant, Department of Orthodontics, University of Rijeka, Faculty of Dental Medicine, Krešimirova 40, 51000 Rijeka, Croatia
(e-mail: tihanazb@fdmri.uniri.hr)

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include the use of antiseptics and fluorides in the daily routine.[3,4] Antiseptics produce an antimicrobial effect by controlling the accumulation of plaque, reducing microorganisms and gingival inflammation, and stimulating healing,[5] while fluorides are very effective cariostatic agents.[6]

Appropriate biomechanics is one of the most important factors supporting effective orthodontic treatment. Changing the mechanical characteristics of the materials used in orthodontic appliances may have an impact on the working properties of appliances and thus influence the clinical progress and outcome. Using fluorides and antiseptics induced the corrosion of dental alloys to some extent, as mainly confirmed under in vitro conditions.[7] Since most research to date has been conducted in vitro, it is expected that in vivo studies will confirm those results. Previous research[8,9] demonstrated the relationship between alloy corrosion and increased surface roughness, which directly affected the working properties of orthodontic wires.

Research results are not straightforward in confirming the effects under in vivo conditions. Fluorides mainly induce general corrosion, while causing pitting corrosion to a lesser extent.[10,11] Antiseptics and probiotics have a small effect on corrosion of nickel-titanium (NiTi) alloys.[12,13] Studies[14] have shown that the best predictor of the NiTi corrosion rate is the concentration of hydrofluoric acid, which is the product of concentration of fluorides and pH. The amount of released Ni and Ti ions during orthodontic treatment generally does not create major clinical problems.[14]

This study aimed to explore whether the commercial agents used in controlling dental biofilm formation and metabolic activity had a significant in vivo effect on the mechanical characteristics of the NiTi alloy used to apply force in an orthodontic appliance system. General corrosion is expected in intraoral conditions. It was hypothesized that the use of fluorides would cause higher corrosion and roughness of NiTi alloy than would chlorhexidine (CHX) or saliva alone. No major changes in the stiffness and hardness of the alloy were expected.

MATERIALS AND METHODS

The sample consisted of 36 subjects, aged 13–42 years, 56% of whom were females. All patients were in orthodontic treatment with 0.022-inch MBT fixed orthodontic appliances (American Orthodontics, Sheboygan, Wisc). After 6 months of leveling and alignment using 0.35-mm (0.014-inch) NiTi archwire and 0.41 $\times$ 0.56-mm (0.016 $\times$ 0.022-inch) NiTi, NiTi archwires with dimensions of 0.508 $\times$ 0.508 mm (0.020 $\times$ 0.020 inch) (BioForce PLUS, Dentsply GAC International, Islandia, New York, USA) were placed intraorally and left in place for 90 days. All NiTi wires were composed of $w(\text{Ni}) = 50.4\%$ and $w(\text{Ti}) = 49.6\%$. The archwires were subjected to testing and analysis. Three experimental groups were formed, with 12 randomly assigned subjects each, according to the randomization plan generated at http://www.randomization.com. All study participants were instructed on the uniform brushing method (Charters technique, three times per day for 2 minutes).

One group consisted of subjects who conducted regular oral hygiene with a toothbrush and toothpaste containing a low concentration of fluoride (1450 ppm), as commonly recommended for patients in orthodontic treatment, for the whole study period of 3 months (the saliva group). The second and the third groups used adjuvant oral hygiene products for the first month: the second group brushed their teeth three times per day without toothpaste and used mouthwash with 0.12% chlorhexidine digluconate (Curasept ADS 212, Curaden, Saronno (VA), Italy) twice per day, while the third group brushed their teeth three times per day, once without toothpaste and twice using gel with 6150 ppm of sodium fluoride (Mirafluor-K-gel, Hager Werken, Duisburg, Germany) for 30 days. For the next 2 months, regular hygiene (Charters technique, three times per day for 2 minutes) with a toothpaste containing a low concentration of fluoride (1450 ppm) was performed.

The local ethical committees approved the research, with written informed consent provided by the subjects (Ethical Committee of the Clinical Hospital Centre Rijeka No. 2170-29-02/1-14-2 and Ethical Committee of the University of Rijeka, Faculty of Medicine No. 2170-24-01-14-05).

Atomic force microscopy (AFM) was used to analyze the surface morphology of NiTi archwires. The measurements of AFM microphotographs were performed on a scanning probe microscope SPM 9600 (Schimadzu, Kyoto, Japan) using a dynamic mode of operation. All samples were scanned at a few points, with the scanning area measuring 30 μm $\times$ 30 μm. The obtained AFM microphotographs were processed and analyzed using the software VectorScan, data processing software for Scanning Probe Microscope (Nanosearch Microscoe, SPM series, OLS/SFT series, Shimadzu Corporation, Analytical and Measuring Instruments Division, Kyoto, Japan, March 2003). Average roughness (R_a), root mean square (R_{ms}), and maximum height (M_h) were estimated. Before the measurements were conducted, each sample investigated, with a length of 5 mm, was fixed on the sample holder using fast-drying glue, and its surface was cleaned with 95% ethanol. Five samples were analyzed.

Fourier Transform Infrared (FTIR) spectroscopy analysis was used to identify the chemical properties

Table 1. Difference in Roughness Parameters Among Experimental Conditions[a]

Experimental Condition	R_a/nm, Mean ± SD	R_{ms}/nm, Mean ± SD	M_h/nm, Mean ± SD
As received	162 ± 52	207 ± 68	1953 ± 1356
Saliva	115 ± 14	147 ± 18	1188 ± 164
Fluoride	84 ± 21	111 ± 22	1067 ± 237
Antiseptic	95 ± 25	124 ± 33	998 ± 390

[a] SD indicates standard deviation.

of the surface. The infrared spectra were recorded using the Nicolet IS35 Thermofisher FTIR-ATR spectrometer (Thermo Fisher Scientific, Waltham, Mass, USA). The FTIR spectrum ranged from 4000 to 500 cm^{-1} at a resolution of 8 cm^{-1}.

Young's modulus of elasticity, hardness, and friction were analyzed using the Nanoindenter G200 (Keysight Technologies Inc, Santa Rosa, California, USA) equipped with a Berkovich testing tip. Samples of archwires were cut to approximately 1.5–2 cm in length and fixed with cyanoacrylate on the sample holder. The depth limit was set at 3000 nm, so calculations of average values were done at depths between 1000 and 3000 nm. Four indents per sample were made. Testing measurements were done on a fused silica sample as a referent. A single-direction wear test with lateral force measurements was used to analyze friction. The normal force was set at 8 mN and the nanoscratch length at 200 μm. Six samples in each experimental group were analyzed.

The corrosion pattern was studied using the electrochemical methods of cyclic polarization and electrochemical impedance spectroscopy (EIS) on a potentiostat Biologic SP-300 (BioLogic Science Instruments, Seyssinet, Pariset, France), as reported previously.[15] The tests were conducted in artificial saliva and compared to as-received archwires with four samples from each experimental group.

A sample size between four and six was calculated to be sufficient, considering a power of 80%; significance of $\alpha = 0.05$; difference in the mean roughness and Young's modulus of 30 between two experimental conditions and standard deviation of 15; difference in the friction of 0.02 and standard deviation of 0.01; and difference in the hardness of 1.5 and standard deviation of 0.9. To explore the differences in the mechanical characteristics between the experimental conditions and the as-received samples, analysis of variance with Student-Newman-Keuls post hoc test was used in commercial software IBM SPSS 22 (IBM Corp, Armonk, NY). Effect size (ie, magnitude of the relationship between the experimental condition and mechanical characteristics) was assessed using $\eta 2$.

RESULTS

Surface Morphology

There were no significant differences in roughness between the experimental intraoral conditions (Table 1; Figure 1). The as-received samples had higher roughness on average but also much higher variability.

Chemical Analysis of the Surface

The infrared spectra of the samples showed characteristic vibration peaks at 1640 cm^{-1} and 1530 cm^{-1} assigned to C=C stretching vibration of the aromatic ring. The absorption peaks in the region 1038–1040 cm^{-1} were assigned to C-O-C stretching vibration of

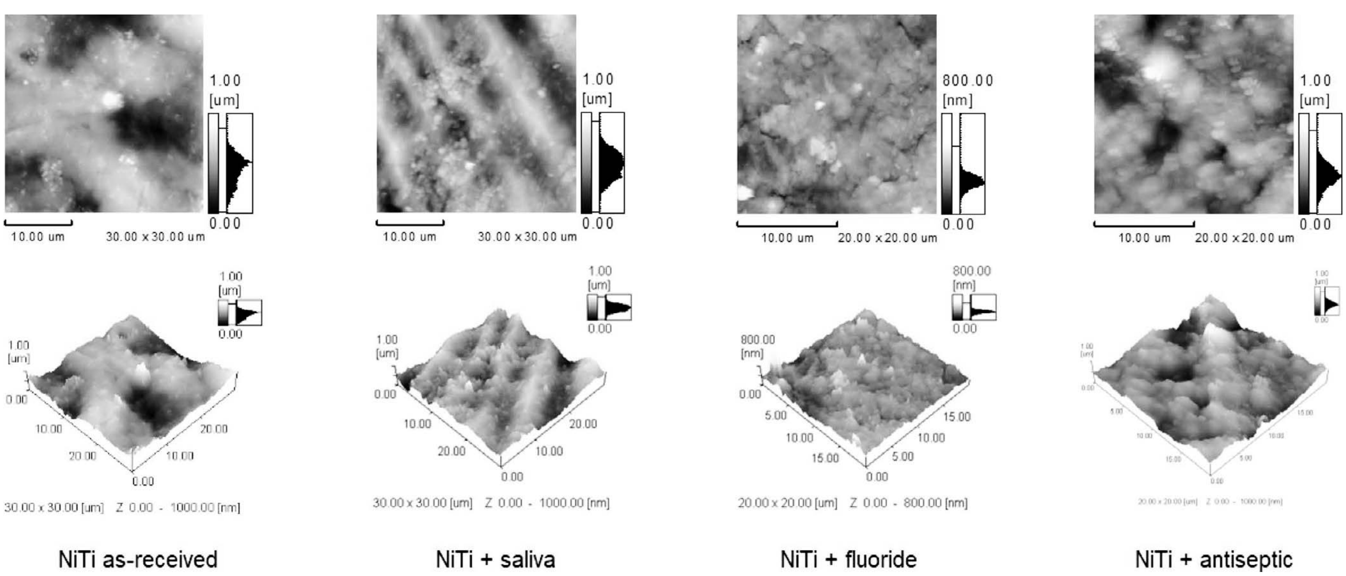

Figure 1. Two- and three-dimensional AFM microphotographs of the studied unexposed and exposed NiTi wire samples.

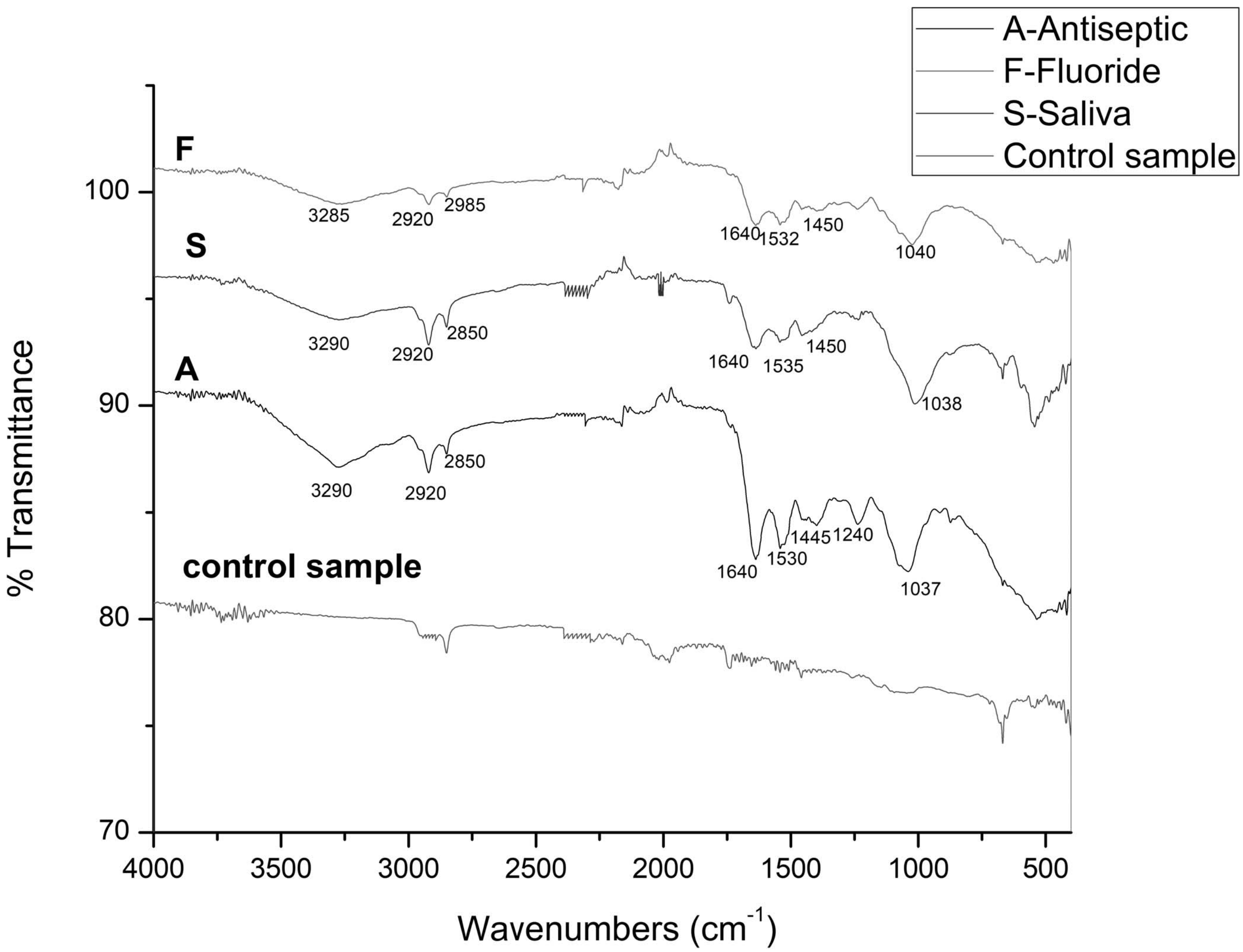

Figure 2. FTIR spectra of samples.

ethers, which indicated the presence of aromatic compound (Figure 2; Table 2). In addition, the absorption peak at 3290 cm^{-1} was attributed to –OH group, and the absorption peaks in the region 1445–1450 cm^{-1} were assigned to $-CH_3$ groups. Two characteristic vibration peaks in the regions 2920 cm^{-1} and 2850 cm^{-1} were assigned to C-H stretching vibration of CH_2 and CH aliphatic and aromatic. All of these characteristic vibration peaks indicated the presence of bisphenol A in the samples analyzed.[16] In the control sample, none of the functional groups that would indicate the presence of an interference compound were identified.

Table 2. Infrared Spectral Data of Archwire Samples

Experimental Condition	Functional Groups, cm^{-1} ν (-OH-)	ν (-C-H-)[a]	ν (-C=C-)[a]	ν ($-CH_3$)	ν (-C-O-C-)
Antiseptic	3290	2920, 2845	1640, 1532	1445	1037
Fluoride	3285	2920, 2850	1640, 1535	1450	1040
Saliva	3290	2920, 2850	1640, 1530	1450	1038

[a] Two characteristic vibration peaks.

Stiffness, Hardness, and Friction

Exposure to intraoral conditions significantly reduced the stiffness and hardness and slightly increased the friction of the NiTi alloy (Figure 3). Fluoride tended to reduce stiffness and hardness more compared to saliva and antiseptics, but not significantly so.

Corrosion Mechanism

Cyclic polarization demonstrated that only some samples exhibited localized corrosion in all media, which is why two graphs are presented with respective results of the samples with and without localized corrosion (Figure 4). The graphs and the corrosion parameters showed that there were no significant differences between the alloys from different media (Figure 4; Table 3). Comparing the results with those

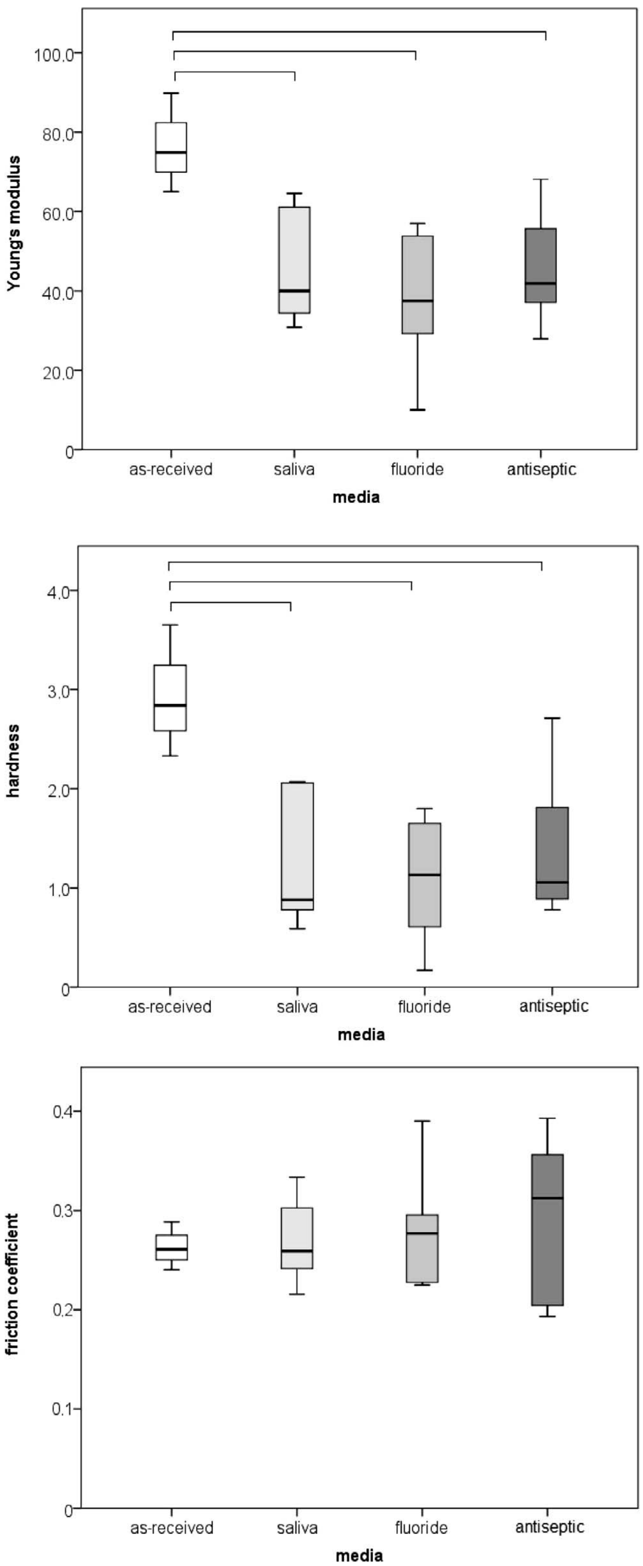

Figure 3. Comparison of stiffness, hardness, and friction among experimental conditions.

Table 3. Comparison of Corrosion Parameters Among Experimental Conditions

Corrosion Parameter	Experimental Condition[a]	Mean ± SD	*P*	η2
Current density, j_{corr}/nAcm^{-2}	Antiseptic	19.6 ± 8.2		
	Fluoride	25.8 ± 9.1		
	Saliva	26.8 ± 7.9		
	As received	31.3 ± 16.3	.530	0.175
Corrosion potential, E_{corr}/mV	Antiseptic	−301.0 ± 23.7		
	Fluoride A	−302.0 ± 11.6		
	Saliva A	−304.3 ± 18.3		
	As received B	76.3 ± 20.4	<.001	0.989
Anodic Tafel slope, b_a/mV dec^{-1}	Antiseptic	220.8 ± 28.9		
	Fluoride AB	286.0 ± 21.7		
	Saliva AB	282.0 ± 16.8		
	As received B	308.3 ± 71.2	.042	0.512
Cathodic Tafel slope, b_c/mV dec^{-1}	Antiseptic A	93.2 ± 6.8		
	Fluoride A	98.0 ± 2.1		
	Saliva A	95.1 ± 4.8		
	As received B	185.3 ± 62.2	.003	0.710
Passive film breakdown potential, E_{bd}/mV	Antiseptic	1010.8 ± 294.3		
	Fluoride	997.5 ± 295.8		
	Saliva	1010.5 ± 313.5		
	As received	1272.7 ± 23.7	.537	0.173
Repassivation potential, E_{rp}/mV	Antiseptic	1182.5 ± 2.1		
	Fluoride	1177.0		
	Saliva	1208.5 ± 38.9		
	As received	1205.0 ± 11.5	.495	0.417

[a] Media that share the same small capital letters do not differ significantly based on Student-Newman-Keuls post hoc test. SD indicates standard deviation.

** *P* level of significance; *** η2: effect size.

obtained from the as-received samples exposed to saliva only, it was seen that exposure to real conditions led to a slight increase in corrosion currents and a shift in corrosion potential in the negative direction. While the tendency toward localized corrosion in the as-received samples was negligible and repassivation was observed in all of the samples, in the case of the wires exposed in vivo, repassivation was observed in only two out of four wires (only one in the case of fluoride). Occurrence of localized corrosion was observed on the sample edges and usually only in the one spot. Such behavior was likely related to the presence of mechanical scratches. For samples exhibiting localized corrosion, E_{bd} was mainly observed around potentials of 0.75 V, while other samples had E_{bd} above 1.2 V. For that reason, the calculated mean E_{bd} values of some samples appeared to be lower than E_{rp} (observed only in samples with E_{bd} value above 1.2 V).

Electrochemical impedance spectroscopy also demonstrated that there were no major differences between the experimental conditions (Figure 5; Table 4). The oxide resistances were higher than in the as-received alloy (exposed to saliva only for 2 hours during measurement). This implied that long-term use

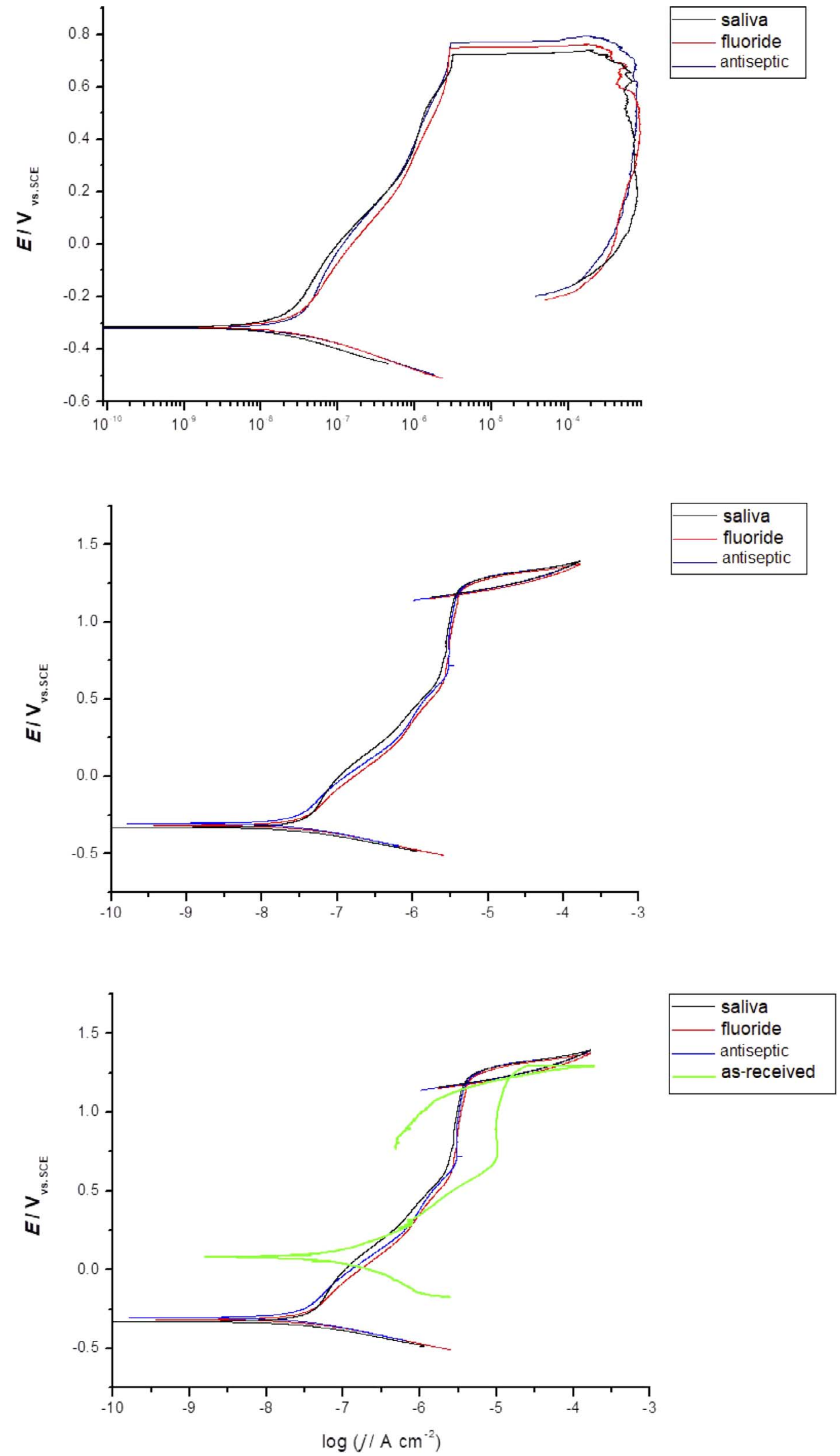

Figure 4. Comparison of NiTi archwires that exhibited pitting corrosion (upper figure), those that did not exhibit pitting corrosion (middle), and comparison with the as-received samples (lower figure).

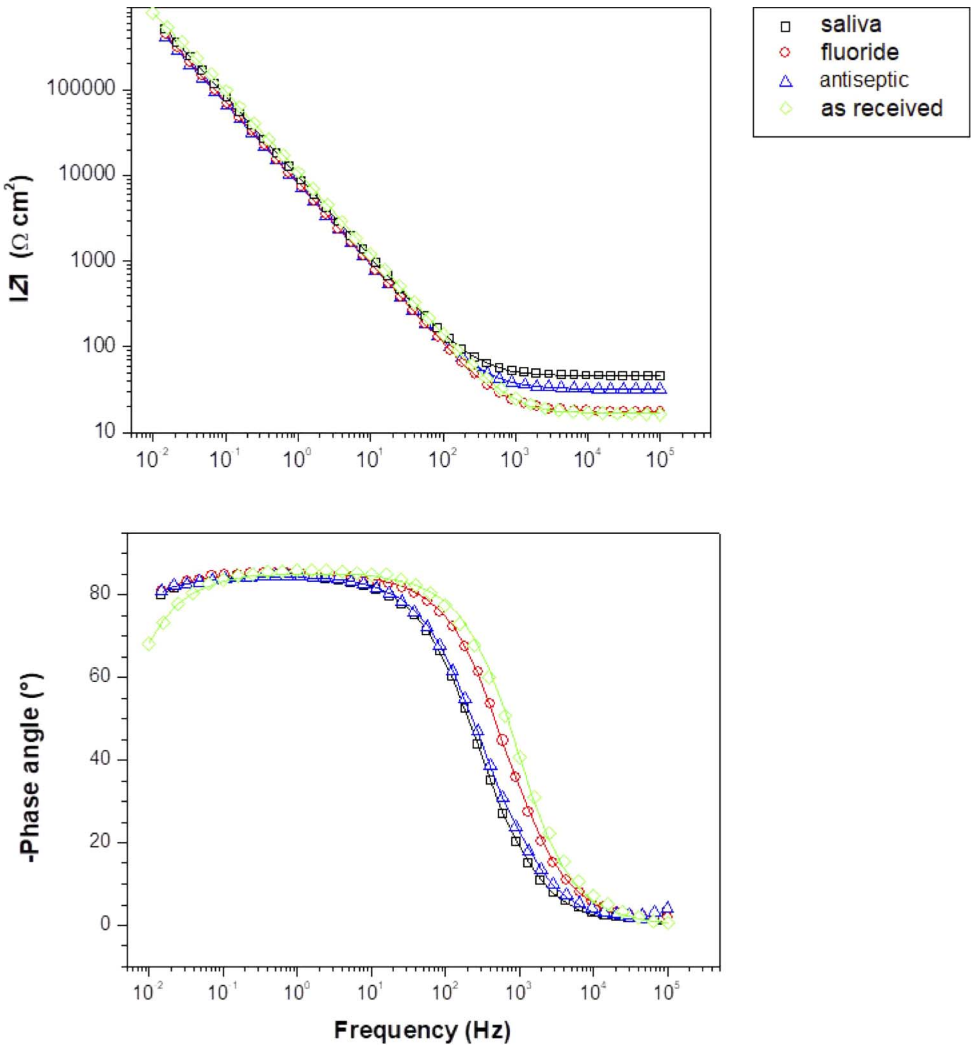

Figure 5. Bode plots for dependence of phase angle on the frequency.

in real conditions of application resulted in formation of the more protective, thicker oxide layer.

DISCUSSION

The present research demonstrated that intraoral exposure of NiTi alloy induced general corrosion, leading to a decrease in some mechanical characteristics. Prophylactic agents did not induce major additional changes. For the first phase of orthodontic biomechanics, it is recommended to use archwires that can produce light, long-term, and constant force values that will not deteriorate by corrosion induced by the use of prophylactic agents.[17]

It seems that preformed NiTi archwires had less homogeneous surfaces than did those that were intraorally exposed. It is possible that intraoral corrosion filled in the porosities, which was evident on archwires exposed to fluorides. Similar findings were reported earlier.[18] It was also possible that corrosion and friction lead to the dissolving/breaking of the tips of the bumps on the wire, which made it less rugged. Other authors[19] reported higher homogeneity for the as-received archwires, while there were heterogeneous surfaces with craters and bumps after clinical use. Some research results[20] implied that lower fluoride concentrations (<2500 ppm) did not increase surface roughness to a large extent in comparison to a high fluoride-containing environment (17,000 ppm).

Bisphenol A (BPA) was detected in all intraorally exposed samples, likely released from orthodontic adhesives, indicating that intraoral exposure to saliva alone also caused its release.[21] No previous research reported any influence of BPA on orthodontic alloys. BPA is used in containers that store food and beverages and for coating the inside of metal products and likely does not have any side effects on metal surfaces.[22,23] It is assumed that BPA has no side effect on the properties of NiTi alloys. Coating of NiTi with

Table 4. Comparison of Impedance Parameters (EIS) Among Experimental Conditions

Corrosion Parameter	Experimental Condition[a]	Mean ± SD	*P*	η2
Electrolyte resistance, R_e/W cm^2	Antiseptic	21.5 ± 7.4		
	Fluoride	20.3 ± 5.1		
	Saliva	23.5 ± 5.1		
	As received	17.5 ± 2.7	.571	0.161
Porous coating/film capacitance, Q1/mS s^n cm^{-2}	Antiseptic	15.420 ± 0.911		
	Fluoride	16.143 ± 1.517		
	Saliva	15.145 ± 0.903		
	As received	.	.476	0.152
n_1	Antiseptic	0.945 ± 0.006		
	Fluoride	0.950 ± 0.000		
	Saliva	0.948 ± 0.005		
	As received	.	.323	0.222
Resistance of porous coating/film, R_1/W cm^2	Antiseptic	0.070 ± 0.031		
	Fluoride	0.130 ± 0.060		
	Saliva	0.116 ± 0.085		
	As received	.	.402	0.183
Surface oxide capacitance, Q_2/mS s^n cm^{-2}	Antiseptic	5.183 ± 0.674		
	Fluoride A	4.300 ± 0.927		
	Saliva A	4.623 ± 1.329		
	As received B	1.793 ± 0.175	.003	0.702
n_2	Antiseptic	0.933 ± 0.005		
	Fluoride	0.943 ± 0.015		
	Saliva	0.935 ± 0.006		
	As received	0.947 ± 0.006	.201	0.333
Compact surface oxide resistance, R_2/W cm^2	Antiseptic	5575.5 ± 588.4		
	Fluoride	5535.3 ± 1306.1		
	Saliva	5512.8 ± 2501.2		
	As received	2381.0 ± 571.4	.056	0.484

[a] Media that share the same small capital letters do not differ significantly based on Student-Newman-Keuls post-hoc test. SD indicates standard deviation.

** *P*: level of significance; *** η2: effect size.

epoxy resin containing BPA improves corrosion resistance and hydrolysis resistance.[16]

NiTi alloys have good corrosion stability based on the passive protective film on their surface. The protective film is composed mainly of titanium oxide (TiO_2) and a small amount of nickel oxide (NiO).[24] Low pH values produced by bacteria in mature biofilm can accelerate corrosion by dissolving the protective layer of the NiTi surface.[25,26] The corrosion resistance of NiTi alloys decreases with applications of fluorides as a result of the deterioration of the TiO_2 surface layer.[15] Nevertheless, bending stress is also a source of corrosion in NiTi wires, although localized.[27,28]

The intraoral environment degrades some mechanical properties of NiTi alloy, and the present study showed that these degradations primarily affect its stiffness and hardness. It appeared that fluorides and CHX did not induce an additional decrease in archwire stiffness and hardness in comparison to saliva alone. Fluorides are a slightly more aggressive medium, and their effect on other types of titanium-based orthodontic archwires has also been documented previously.[29] Some in vivo studies[30] reported that fluoride agents could decrease the elasticity of NiTi wires and, consequently, prolong orthodontic treatment. Friction was also found to slightly increase after intraoral exposure, but not significantly. Some in vitro studies[31] also confirmed that even low saliva pH, such as 4.8, did not increase the friction of NiTi alloy to a large extent. Similar results were reported[32] after comparison of in vitro and intraoral fluoride application. Several factors could diminish the effect of fluorides intraorally, namely, dilution and buffering by saliva and rapid formation of biofilm on the material surface.[32] There is a high correlation between friction and roughness, as reported earlier, with lower roughness related to lower friction.[33]

NiTi alloys do corrode during intraoral exposure, primarily because of the influence of saliva, but they do not corrode significantly more if fluorides and antiseptics such as CHX are used. Fluoride solutions induce the destruction of the titanium oxide layer of the alloy, resulting in the formation of titanium fluoride, titanium oxide fluoride, or sodium titanium fluoride on the surface.[32] Titanium has a high affinity to hydrogen, which may be introduced over time through the corrosion process, leading to hydrogen-induced cracking of the NiTi alloy. The type of corrosion at issue is mainly general corrosion, but pitting corrosion can also occur, independent of the medium. In vitro research[34] reported that fluorides caused localized, pitting corrosion on NiTi alloys and, with lower pH of fluoride solution, produced higher corrosion. Localized corrosion would occur primarily if there were inhomogeneity in material composition or if the protective passive layer was damaged. Nevertheless, in vitro research tends to oversimplify and overestimate the influence of fluorides in the oral environment, including hydrogen embrittlement as a mechanism related to archwire fractures.[35]

This study simulated real conditions, since the use of CHX is not recommended for more than 1 month because of side effects (staining of teeth, tongue, and appliances and changes in taste), while high concentration fluorides are not appropriate for long-term use because they introduce a risk of fluorosis. The shortcoming of this study was that the toothbrush that participants used was not standardized, so characteristics of the toothbrush as well as the lubricating effect of the toothpaste could have affected the archwire roughness.

CONCLUSIONS

- Intraoral use of antiseptics and fluorides produces primarily general corrosion of NiTi alloy and reductions in NiTi stiffness and hardness, but it causes no major changes in roughness and friction.

- Fluorides and the antiseptic CHX do not increase corrosion more than saliva itself, nor do they further modify the mechanical properties of the NiTi alloy.

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