Original Article

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Nickel and copper ion release, deflection and the surface roughness of copper-nickel-titanium orthodontic archwire in sodium fluoride solution

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Abstract

OBJECTIVE: Sodium fluoride (NaF) is commonly used in oral hygiene products, leading to corrosion and reduced archwire properties. In addition, ion release can cause allergies and become toxic to the oral environment. This research aimed to observe the Nickel (Ni) and Copper (Cu) ions released that affected initial corrosion as deflection and surface roughness changed in the Copper-Nickel-Titanium (CuNiTi) archwire.

MATERIAL AND METHODS: The total samples were 54 copper-nickel-titanium (CuNiTi-Tanzo, American orthodontic®) archwires immersed in three solutions. Artificial saliva was used in the control group NaF 0.05%, and a NaF 0.15% solution was used in the intervention groups ($n = 6$). The groups were divided into three observation times (two, four, and six weeks). Cu and Ni ions released, deflection, the surface roughness of the archwires, and solution acidities were recorded and analyzed.

RESULTS: Ni and Cu ion release and surface roughness of the CuNiTi archwires significantly increased as the NaF concentration increased. The Ni ion release improved along the immersion period; the opposite was true for the Cu ion release. The solutions became more alkaline after the CuNiTi archwires were immersed. The pH and the archwires' deflections of the three solutions did not show significant differences.

CONCLUSION: The NaF increased Cu-Ni ion release and surface roughness but not the deflection force of the CuNiTi. The increase was affected by the concentration and duration of immersion.

Keywords:

Archwire deflection, copper ion, CuNiTi archwire, ion release, nickel ion, sodium fluoride, surface roughness

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Introduction

Copper-Nickel-Titanium (CuNiTi) orthodontic archwire was introduced in the 1990s as a new type of nickel-titanium alloy.[1,2] The addition of copper (Cu) causes a more constant force distribution during activation because the loading and unloading forces are less than that of the NiTi orthodontic archwire. CuNiTi archwire is superior to NiTi archwire in temperature control, springback, and deformation resistance.[3,4]

The general composition of CuNiTi archwire is 42.99% titanium, 49.87% nickel, 0.50% chromium, and 5.64% copper.[5] These components, when exposed to the oral environment, undergo corrosion due to the ion release caused by chemical, mechanical, thermal, microbiological, and enzymatic changes.[6] Some ions released by the orthodontic archwire – Ni and Cu, for instance – present adverse effects on oral mucosal surfaces. Ni can cause hypersensitivity and allergic reactions, particularly contact dermatitis and systemic

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contact dermatitis.[7] Cu can induce cell toxicity when the concentration exceeds the recommended level.[8] Corrosion also increases the surface roughness of the orthodontic material and degrades the quality of the archwire properties when unloading the force on the teeth.[9] By adding frictional force as the contact areas between the archwire and bracket could be increased and affected the efficiency of orthodontic tooth movement.[10]

Fixed orthodontic usage can increase plaque retention and cause difficulties in maintaining oral hygiene.[11] For this reason, orthodontists have suggested patients use mouthwash as a coadjuvant with toothpaste for plaque control.[12] Sodium fluoride (NaF) can often be found in toothpaste and mouthwash. Despite its anticaries effect, NaF can affect CuNiTi archwire corrosion resistance.[13] A higher NaF concentration leads to a decrease in the corrosion resistance of the archwire. Microscopic photoanalysis of NiTi and CuNiTi immersed in low-concentration NaF showed localized pitting and colorization, whereas general and extensive pitting was observed in high-concentration NaF.[14] NaF can also alter the mechanical properties of an archwire; the concentration, duration of exposure, and fluoride's acidity cause a decrease in archwire deflection, resulting in a longer unloading process of the archwire for tooth movement.[3,15-17] Nickel ion released by NaF exposure was already well known to cause corrosion of the orthodontic archwire. Still, the Cu ion released also plays quite an essential role in giving CuNiTi archwire its superior properties. This study aimed to quantify and see if the NaF mouthwash induces the Cu ion release and if the Cu ion release has the same quantity as the Nickel ion released, which could degrade the unloading force to the deflection of the archwire.

Material and Methods

This was experimental laboratory research to analyze the Ni and Cu ion release, the unloading force from deflection, and the surface analysis of the CuNiTi archwires. The study was approved with ethical clearance No. 347/KEP/USU/2021 by the Health Research Ethics Committee (KEPK) of Universitas Sumatera Utara.

Groups and Sample Preparation

The samples consisted of 54 CuNiTi Tanzo™ archwires (American Orthodontics, Sheboygan, WI, USA), 0.016 × 0.025 inches and 4-cm long. The samples were divided into three groups (n = 18). The control group was Group A, NaF 0.05% was Group B, and NaF 0.15% was Group C. Each group was further divided into three subgroups (n = 6) based on observation times of two, four, and six weeks at 37°C.

The NaF solution was custom-made by the author. The NaF 0.05% solution was obtained by dissolving 0.09-gram of NaF powder (Merck KGaA, Darmstadt, Germany) into 180 mL of artificial saliva solution and 0.27-gram of NaF powder into 180 mL of artificial saliva solution for 0.15% NaF solution. In Group A, the archwires were immersed in 20 mL of artificial saliva ($NaHCO_3$ 9,8%, Na_2HPO_4. $12H_2O$ 9,3%, NaCl 0,47%, KCl 0,57%, $CaCl_2$ or $CaCl_2$. $2H_2O$ 0,04% (0,045%), MgCl or $MgCl_2.2H_2O$ 0,06% (0,065%)) for two, four, and six weeks. In Groups B and C, archwires were first immersed in 10 mL of artificial saliva. Before two, four, and six weeks ended, 10 mL NaF 0.05% (Group B) or 10 mL 0.15% (Group C) was added into the solution, and archwires were immersed for 28, 54, and 84 minutes in the combined solutions. The 28, 54, and 84 minutes immersion times were chosen to represent the daily use of NaF in oral hygiene routines with two-week intervals before the dental checkup during orthodontic treatment.

Nickel and copper ion release

A digital pH meter measured solution acidity before and after archwire immersion (Hanna, HI98107). In addition, Ni and Cu ion releases from the sample solutions were analyzed at Balai Standardisasi dan Pelayanan Jasa Industri (Baristand) Medan using atomic absorption spectrometry (AAS, Shimadzu AA7000).

Deflection and surface roughness

The CuNiTi archwires were tested for deflection with the three-point bending method using the Universal Testing Machine (Tensilon, RTF-1350, Japan) in the Impact Fracture Research Center Laboratory, Faculty of Engineering, Univeristas Sumatera Utara. Three archwires from each subgroup were taken to be tested for surface roughness using a scanning electron microscope (Hitachi TM3000), with 2000 × magnification, conducted in the Research Center Laboratory, Faculty of Mathematics and Science, Univeristas Sumatera Utara.

Statistical analysis

The data results were statistically analyzed with SPSS 26.0 (Chicago, USA). The normality test was carried out using the Shapiro–Wilk test. Data that were not normally distributed were analyzed with the Kruskal–Wallis test, while normally distributed data were tested with ANOVA GLM-RM. The results were significant if the P-value was below 0.05.

Results

The acidity (pH) data and Ni ion release were not normally distributed, while Cu ion release and deflection data were normally distributed. The pH of the three sample groups became more alkaline than the baseline pH over time [Table 1 and Figure 1]. The pH was significantly different among different observation periods, except in NaF 0.05% group. Ni ion release increased over time in all groups [Table 2 and Figure 2], while Cu ion release decreased over time in all groups [Table 3 and Figure 3]. All groups showed significantly different Ni and Cu ion mean values over time ($P < 0.05$). Samples immersed in NaF 0.15% led to the most significant value in both Ni and Cu ion releases.

Based on the Kruskal–Wallis test, there were significant differences in the unloading force overtime only in NaF 0.15% group ($p < 0.05$) [Table 4 and Figure 4]. The significant difference was only shown in week 4 ($p < 0.05$). The scanning electron micrograph showed the surface roughness of the three sample groups in three immersion periods. The micrograph could only be subjectively analyzed, and the exact quantitative result could not be provided. However, it was evident that the samples immersed in NaF 0.15% displayed the most defects and porosities among the three sample groups [Figure 5].

Table 1: Acidity (pH) solution between groups and subgroups (*n*=6). The baseline pH was 7.5

Solution	Week 2		Week 4		Week 6		*P*
	Med	IR	Med	IR	Med	IR	
Artificial saliva	8.30	0.06	8.13	0.54	8.03	0.12	0.03*
NaF 0.05%	8.10	0.20	8.17	0.73	7.96	0.14	0.163
NaF 0.15%	8.26	0.56	8.17	0.35	8.15	0.08	0.027*
*P**	0.004*		0.663		0.054		

Med=Median, IR=Interquartile range, *significantly different with $P<0.05$ by Kruskal–Wallis test

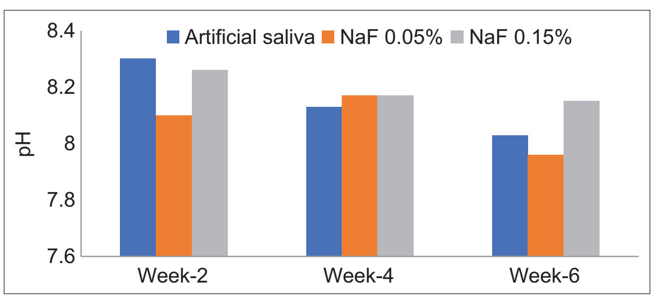

Figure 1: Acidity (pH) solution between groups and subgroups (n = 6)

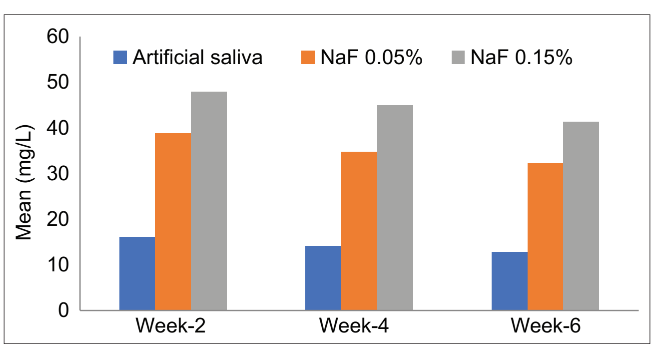

Figure 3: Values of Cu ion release between sample groups and subgroups

Discussion

Orthodontic material biocompatibility has been a significant concern for clinicians due to the long treatment period and high incidence of allergic reactions.[18] Ni-containing alloy has remained controversial because Ni has superior physical properties but can exhibit adverse effects like contact dermatitis.[19] While studies

Table 2: Description values of Ni ion release between sample groups and subgroups

Solution	Week 2 (μg/L)		Week 4 (μg/L)		Week 6 (μg/L)		*P*
	Med	IR	Med	IR	Med	IR	
Artificial saliva	49.50	5.0	99.50	4.0	128.50	3.0	0.001*
NaF 0.05%	58.00	4.25	100.00	2.5	133.00	0.25	0.001*
NaF 0.15%	100.50	4.5	128.50	3.0	168.00	1.8	0.001*
*P**	0.001		0.002		0.001*		

Med=Median, IR=Interquartile range, *significantly different with $P<0.05$ by Kruskal–Wallis test

Table 3: Description values of Cu ion release between sample groups and subgroups

Solution	Week 2 (μg/L)		Week 4 (μg/L)		Week 6 (μg/L)		*P*
	Mean	SD	Mean	SD	Mean	SD	
Artificial saliva	16.03	0.45	14.04	0.56	12.81	30.30	0.001*
NaF 0.05%	38.73	0.68	34.69	1.41	32.15	0.46	0.001*
NaF 0.15%	47,86	0.86	44.92	0.89	41.29	0.56	0.001*
*P**	0.001*		0.001*		0.001*		

*ANOVA test with a significance value of $P<0.05$

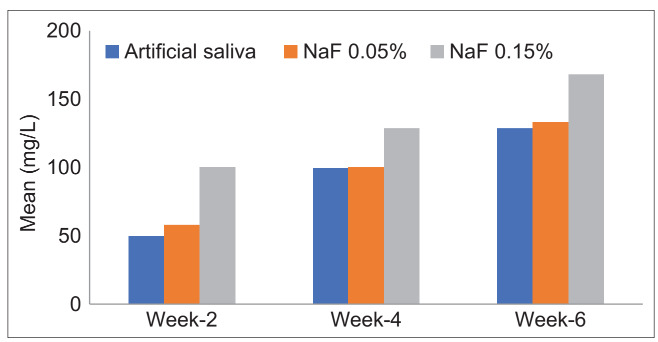

Figure 2: Values of Ni ion release between sample groups and subgroups

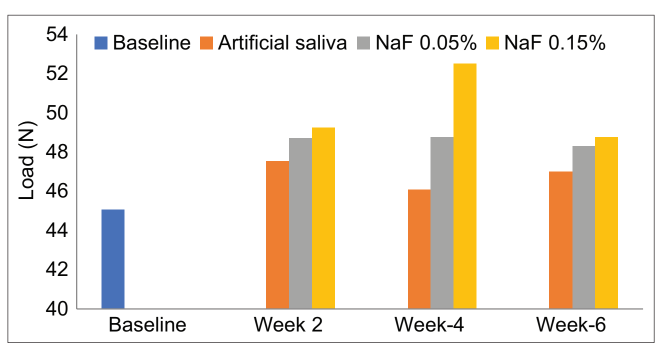

Figure 4: Values of CuNiTi archwire deflection between sample groups and subgroups

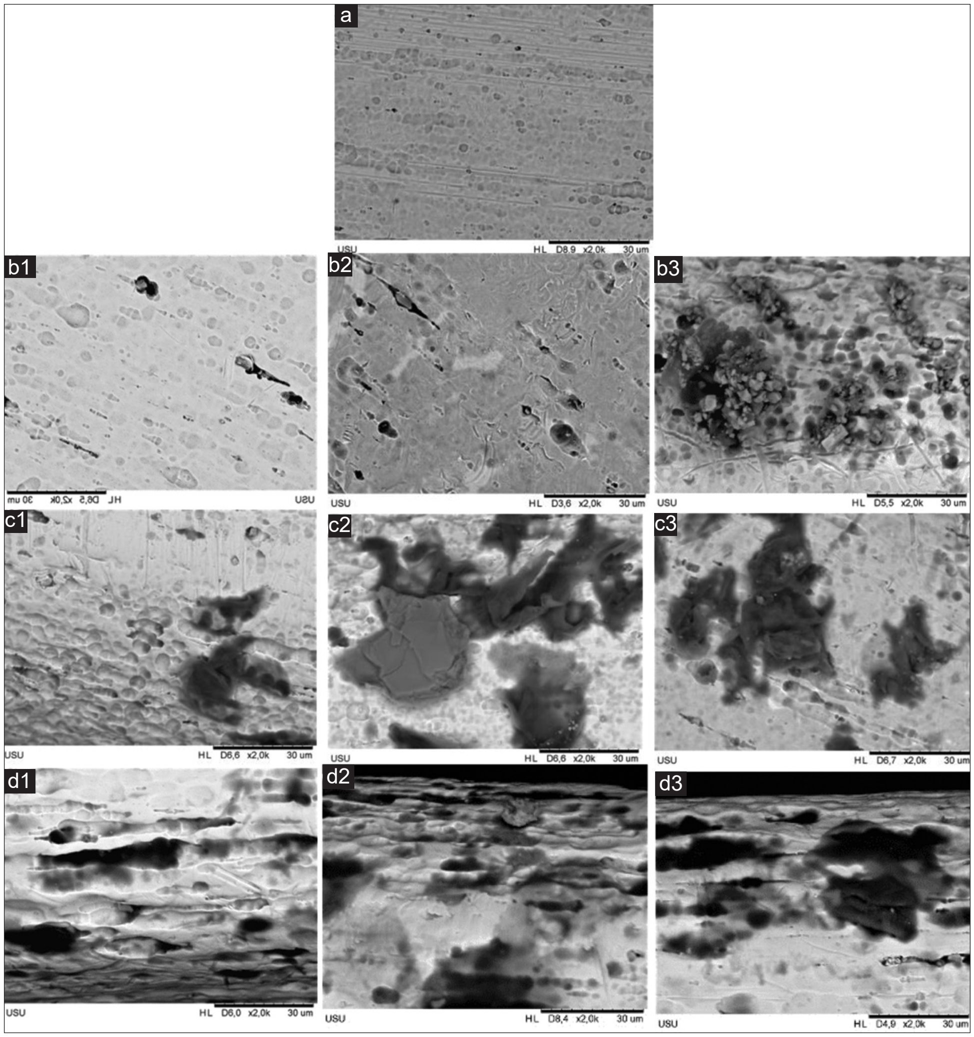

Figure 5: Surface analysis of CuNiTi archwire. (a) Baseline. (b) Control group (artificial saliva) (c) 0.05% NaF group (d) 0.15% NaF group; (subscripted 1, 2, 3 means 2, 4, and 6 weeks)

Table 4: Description values of CuNiTi archwires' unloading force between the baseline value of 45.049 Newton (N)

Solution	Week 2 (N)		Week 4 (N)		Week 6 (N)		*P*
	Med	IR	Med	IR	Med	IR	
Artificial saliva	47.52	3.61	46.08	3.11	46.99	3.81	0.262
NaF 0.05%	48.71	2.85	48.75	1.85	48.30	2.31	0.623
NaF 0.15%	49.24	5.34	52.49	1.39	48.76	2.51	0.019*
*P**	0.372		0.001*		0.343		

Med=Median, IR=Interquartile range, *Significantly different with $P<0.05$ by Kruskal–Wallis test

about Cu ion effects were limited, previous studies stated copper oxide nanoparticles were cytotoxic toward cell membrane and mitochondria function, and Cu ions became cytotoxic at 10 ppm.[20,21]

This study showed that immersion solutions were important in determining ion release and deflection. The samples were most affected by NaF 0.15% solution, as ion release was the highest in this group. The results of this study were aligned with a previous study by Jafari *et al*.[22] about the immersion of Ni-Cr alloys in two different types of mouthwash; the study showed that Ni ion release was higher from samples immersed in NaF mouthwash than from samples dipped in non-NaF mouthwash. These results were similar to another study by Lee *et al*.[23] that evaluated the effect of fluoride prophylactic agents on NiTi wires and showed that corrosion resistance decreased as NaF concentration increased. NaF in products containing fluoride reacts with hydrogen ions and forms hydrofluoric acid, which dissolves the protective oxide layer on the surface of orthodontic components and causes corrosion.[24]

Immersion duration could also affect archwire ion release. This study presented different results for Ni and Cu ions. Ni ion release increased over time. This result was in line with research by Senkutvan *et al*.,[25] which showed a significant increase in Ni ion release from CuNiTi archwires with three immersion periods (one, two, and three weeks) – the highest was in the third week. Habar and Tatengkeng[26] also did similar research using 33 and 66 days of immersion. Their result also displayed a significant increase over time. Chloride ions in electrolyte solutions can damage the oxide layers on the wire surfaces, resulting in metal ion release.

There was a significant decrease in Cu ion release over time. The result was comparable to the study by Freitas *et al.*[27] regarding the release of Cu, Zn, and Ag ions in orthodontic appliances placed in oral cavities for various durations. Their study showed that Cu ion release decreased gradually. Biofilm formation caused chemical passivity, which changed the characteristics of the alloy surfaces.

The difference in unloading force at week four in this study showed the alteration of mechanical properties after immersion. A previous study by Ahrari *et al.*[3] showed a significant decrease in CuNiTi unloading force after one day of immersion in 0.2% NaF solution. Even so, in this study, the unloading force of the wire immersed in NaF solutions increased. Similar results of increasing unloading force were reported by Pop *et al.*,[28] where NiTi wires were immersed in fluoride and coke solution, and by Aghili *et al.*,[29] where they were immersed in mouthwash solution. This difference did not mean mechanical properties improved after immersion; it was due to the different methods and materials used.[28] The significant difference in the week 4 group could be the reason for the regular visits of orthodontic patients, which are usually scheduled at four- to six-week intervals.[30,31]

Surface roughness analysis showed an increase in wire degradation coinciding with the immersion durations and fluoride concentration. Greater surface roughness results in a higher corrosion rate, causing material failure.[32,33] Orthodontic wire is manufactured with a passive layer that is resistant to corrosion, but it could be broken after frequent exposure to corrosive agents. Titanium-based alloy could corrode after prolonged contact with fluoride agents in both acidic and alkaline environments, thus making surface pitting corrosion in CuNiTi due to microcavities.[6,17,24] Fatene *et al.*[14] reported that CuNiTi did not have better corrosion resistance than NiTi archwire in fluoride solution. The artificial saliva used as an immersion solution in this study consisted of NaCl, which influenced higher corrosion on orthodontic appliances, and sodium bicarbonate, which is known to increase salivary pH value.[34] Chloride ions damage the protective layer of orthodontic appliances and release ions from the archwire, making the environment more alkaline and further triggering corrosion.[35] Mohammed *et al.*[36] reported similar results after immersion of CuNiTi in artificial saliva, and the surface roughness was higher compared to that of the non-immersed group.

Our study showed that the daily use of NaF would lead to mechanical degradation, such as ion release and wire property alteration, which might affect the lifetime of wire usage and function. The results of this study were limited to showing ion release and changes in the deflection force on wires of the American Orthodontics brand measuring 0.016 × 0.025 inches. The results of the study were also influenced by the presence of uncontrollable variables such as air humidity and the wire production process, confounding factors such as not changing the solution during immersion, and other factors such as artificial saliva composition, fluoride concentration, and pH level. The result of ion release was also tested using atomic absorption spectroscopy (AAS) instead of inductively coupled plasma optical emission spectroscopy (ICP-OES) even though it is known to be more sensitive and can measure more than one element at a time, but laboratory conditions did not allow analysis using ICP-OES when immersion was carried out, hence, the use of AAS.

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Conflicts of interest

There are no conflicts of interest.

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