Release of nickel ion and surface microstructure of NiTi archwire after immersion in tomato and orange juice

H F Lubis* and Y A Purba
Department of Orthodontics, Faculty of Dentistry, Universitas Sumatera Utara, Medan, North Sumatra 20155, Indonesia
*Email: hildadrgusu@gmail.com

Abstract. One of wires commonly used in orthodontic treatment is nickel-titanium (NiTi) archwire. NiTi archwire has the potential to release ions that can cause allergic and cytotoxic reactions. This study aimed to specify the difference in the amount of nickel ion release and surface microstructure of NiTi archwires after immersing in tomato and orange juice. NiTi archwire with a diameter of 0.016 inches and a length of 5 cm was used as the sample, which was immersed in 15 ml of solution and then stored at 37°C in an incubator for 24 hours. The samples were divided into two treatment groups (immersed in tomato and orange juice), each with nine samples. The immersion solution was tested for ion release using an Inductively Coupled Plasma–Mass Spectrometer. The microstructure of the wire surface was examined using a scanning electron microscope (SEM). The results revealed that group 1 has higher average amount of nickel ion release than group 2 and control. SEM result showed that the surface microstructure of the NiTi archwire in group 1 is roughest. There was a significant difference between the amount of nickel ion released and surface microstructure on NiTi archwire after being immersed in tomato and orange juice.

1. Introduction

Treatment with fixed appliances is the most commonly used orthodontic treatment [1]. The main components of a fixed orthodontic appliance are the bracket, band, ligature, and archwire [2]. The orthodontic wire is made up of various alloys of materials, one of which is nickel-titanium (NiTi) alloy orthodontic wire [3]. NiTi wire is the material of choice for orthodontic applications because of its shape memory effect and superelasticity [4]. Although NiTi wire has a high nickel content, the nickel release from these wires is low, and well below the threshold needed to lead a biological effect (0.5 m/cm2). Corrosion resistance is affected by environmental factors such as pH, temperature, and so on [5]. NiTi wire, which is used in fixed orthodontic appliances, is always in contact with oral tissues. The presence of low pH and high temperature in the oral cavity increases corrosion and nickel ion release several times more than the presence of high pH and low temperature in the oral cavity [6].

Consuming healthy foods and beverages, such as fruit juices, is important. Fruit juices are popular due to their nutritional value, minerals, and vitamins [7]. The pH of tomatoes (around 4 – 4.5) and oranges (below 3.7) is lower than the critical enamel pH of 5.5 [8-10]. Acidic pH, as well as the presence of aggressive ions such as chloride ions, can speed up the loss of the protective coating on the wire and lead to corrosion [11]. Corrosion is the result of a metal's reaction with its surroundings, causing it to corrode. Metal ions generated during the corrosion process can have an impact on the tissues around the mouth cavity, as well as other organs further away. Toxic and allergic reactions can arise as a result of the reaction. Corrosion in the oral cavity will attack the orthodontic wire surface,
causing ions to be released, causing the wire surface to become porous. The more ions released, the more porous the wire's surface becomes, and as a result, the wire's surface seems rougher [3,12-13].

Sharma et.al [11] investigated the effect of some fruit juices and chloride ions on the corrosion activity of orthodontic wires in their study. According to his studies, tomato juice is the most damaging to the surface of stainless steel orthodontic wires when compared to other fruits. When compared to soft beverages, Marcin et.al [14] discovered that orange juice had no significant influence on the discharge of nickel ions from the wire, band bracket. Fruit juice consumption rises as people become more aware of the benefits of living a healthy lifestyle. Therefore, this study aimed to see if there were any differences in the release of nickel ions and the surface of NiTi archwire after being immersed in tomato and orange juice.

2. Materials and Methods
This is a laboratory experimental study with post-test only control group design. A NiTi archwire with a diameter of 0.016 inches and a length of 2 cm was used as the sample. The sample was divided into two treatment groups and one control group, each group with nine samples. The first group was NiTi archwire immersed in tomato juice, the second in orange juice, and the control group in artificial saliva.

The samples were immersed in tomato, orange juice, and artificial saliva. Tomato and orange juice are processed using a juicer that has been cleaned and cut beforehand. NiTi archwire was put into a Petri dish containing an immersion solution based on grouping and placed in an incubator at 37°C for 24 hours. After 24 hours, samples were taken from the incubator and separated from the solution. The solution’s pH was measure before and after immersion. The release of nickel ions from the immersion solution was measured using an Inductively Coupled Plasma – Mass Spectrometer (ICP-MS) (Figure 1a). A scanning electron microscope (SEM) was used to examine wire samples to see the microstructure of the wire surface (Figure 1b).

The result of the research were processed statistically using the SPSS version 20. The normality of the research data was determined using the Shapiro Wilk test. If the test results are normally distributed (p>0.05), the test is carried out with the One Way ANOVA parametric statistical test with a 95% confidence level.

3. Results
The average amount of nickel ion release obtained in group 1 was 0.010±0.000 ppm, 0.009±0.000 ppm in group 2, and 0.002±0.000 ppm in the control group, according to the findings (Table 1). Based on these findings, the average amount of nickel ion release in the treatment group (tomato and orange
juice) was higher than in the control group (artificial saliva). The immersion of NiTi archwire in tomato juice resulted in the highest average amount of ion release.

The Shapiro-Wilk statistical test was used to test the data normality, and the results showed that the data distribution was normally distributed with a significance value of p≥0.05. The measurement of the amount of nickel ion release from NiTi archwire in group 1 had a significance value of p = 0.742 (p≥0.05), group 2 had a significance value of p = 0.935 (p≥0.05), and the control group p = 0.067 (p≥0.05). Test was continued using the Levene Test which showed homogeneous data with a significance value of p = 0.128 (p≥0.05). Data analysis was continued by using the Oneway ANOVA test (Table 2). With a significance value of p = 0.001 (p<0.05), the test results revealed a significant difference in the amount of nickel ion released in NiTi archwire after immersion in tomato and orange juice.

| Group | N  | Average amount of ion release (ppm) (X±SD) |
|-------|----|------------------------------------------|
| 1     | 9  | 0.010 ± 0.00                             |
| 2     | 9  | 0.009 ± 0.00                             |
| Control | 9 | 0.002 ± 0.00                             |

Table 1. The average amount of nickel ion release in NiTi archwire after immersing in tomato and orange juice.

| Group | Average (ppm) | Standard Deviation | p     |
|-------|---------------|--------------------|-------|
| 1     | 0.010         | 0.000              |       |
| 2     | 0.009         | 0.000              | 0.001 |
| Control | 0.002 | 0.000              |       |

Table 2. Differences in the release of nickel ions in NiTi archwire after immersing in tomato and orange juice.

Immersion of NiTi archwire in tomato and orange juice causes ion release from the wire surface, causing the microstructure of the wire surface to change. Changes in the surface of the wire can take many different forms, including a rough surface, the presence of cavities, peeling layers, scratches, etc.

Figure 2. SEM image of NiTi archwire after immersing in tomato juice (a) 250x Magnification, (b) 500x Magnification, (c) 1000x Magnification
The results of the SEM analysis on NiTi archwire soaked in tomato juice can be seen in Figure 2. Figure 2a with 250x magnification shows that there are parts on the surface of the NiTi archwire that are smooth and some are abnormal with a rough surface. Figure 2b with 500x magnification shows more clearly the part of the wire surface that is experiencing abnormalities. Seen on the wire that there is a peeled part. Figure 2c with 1000x magnification shows the presence of small cavities and very fine lines in the wire.

![Figure 2. SEM image of NiTi archwire after immersing in tomato juice (a) 250x Magnification, (b) 500x Magnification, (c) 1000x Magnification](image)

**Figure 3.** SEM image of NiTi archwire after immersing in orange juice (a) 250x Magnification, (b) 500x Magnification, (c) 1000x Magnification

The results of SEM analysis on NiTi archwire soaked in orange juice can be seen in Figure 3. Figure 3a with 250x magnification shows that the entire surface of the NiTi archwire is abnormal. Figure 3b with 500x magnification shows that there is a surface of the wire where the coating is peeling off (red arrow) and there is a smooth part (blue arrow). Figure 3c with 1000x magnification shows more clearly the difference between the part that is peeled off and the part that is not. Figure 3c shows the absence of voids in the wire and the presence of very fine lines.

![Figure 3](image)

**Figure 4.** SEM image of NiTi archwire after immersing in artificial saliva (a) 250x Magnification, (b) 500x Magnification, (c) 1000x Magnification

The results of SEM analysis on NiTi archwire immersed in artificial saliva can be seen in Figure 4. Figures 4a and 4b with 250x and 500x magnification do not show very clear abnormalities on the entire surface of the NiTi archwire. Figure 4c with 1000x magnification shows the presence of fine lines on the surface of the wire.

4. Discussion

In this study, NiTi archwire was immersed in tomato juice, orange juice and artificial saliva as a control and then calculated nickel ions released in tomato juice (pH before and after immersion is 4.7), orange juice (pH before and after immersion is 4, 9) and artificial saliva (pH before and after immersion is 8.1). The average result of measuring the amount of nickel ion released in NiTi archwire after soaking in tomato juice is 0.010 ppm, in orange juice is 0.009 ppm and in artificial saliva is 0.002 ppm (Table 1). According to the findings of this study, group 1 had the highest average amount of nickel ion release after immersing in tomato juice.
The findings of this study are consistent with the findings of Sharma et al. [11] who found that when nickel ions were released on stainless steel orthodontic wires that were immersed in various types of juice, including tomato and orange juice, the tomato juice caused the most corrosion. This study supports the findings of Kristianingsih et al. [15] who found that when stainless steel orthodontic wires were immersed in carbonated drinks and artificial saliva, and the result showed that the average value of the release of Ni and Cr ions in carbonated drinks was higher than in the untreated group. This is also in line with the findings of Situmeang et al. [16] who compared stainless steel orthodontic wires immersed in artificial saliva containing vinegar (pH 2.96) to artificial saliva that did not contain vinegar (pH 6.8). Following that, his research discovered a significant difference in nickel ion release between the two groups.

Citric acid, malic acid, tartaric acid, succinic acid, and benzoic acid are just a few of the organic acids found in tomatoes and oranges, all of which contribute to the acidic pH of these two fruits. The citric acid (C6H8O7) contains a lot of H+ ions, which can speed up corrosion. Vitamin C (ascorbic acid) is found in both of these fruits, and it can speed up corrosion by destroying the passive layer. The average number of ions released in NiTi archwire immersed in tomato juice was higher in this study. According to previous research, this occurs because tomato juice has higher acidity than orange juice and artificial saliva [17,18].

There is a passive film on the outside of the NiTi archwire. Nickel ions will be released onto the wire's surface if the passive layer is damaged. One of the causes of damage to the passive layer is an acidic pH. In addition to causing damage to the passive layer on the metal surface, more acidic conditions (low pH) cause an increase in the concentration of H+ ions, which is corrosive and increases the amount of metal ion release. When the concentration of H+ ions rises, more H+ ions have reduction reactions, causing more metal ions to oxidize, speeding up corrosion [16,19-20].

SEM results of untreated NiTi wire showed a smooth wire surface, no peeling layer, no voids but very fine lines (Figure 5). The SEM results of wire soaked in tomato juice showed the appearance of peeling parts, small cavities and very fine lines on the wire (Figure 2). The SEM results of NiTi wire soaked in orange juice show a picture of the surface of the wire with peeling layers all over the surface and the presence of very fine lines (Figure 3). SEM results of NiTi wire immersed in artificial saliva showed a smooth surface, but there were very fine lines on the wire surface (Figure 4).

![Figure 5](image_url) SEM image of untreated NiTi archwire (a) 250x Magnification, (b) 500x Magnification, (c) 1000x Magnification

SEM results of untreated and treated NiTi wire demonstrated that the release of ions from NiTi wire caused damage to the wire surface. The damage that occurs to the NiTi wire immersed in tomato juice is more severe than that which occurs in orange juice. The damage that occurs in tomato juice but damage has already caused localized peeling layer is thick and the small cavity whereas, the damage spreads orange juice but not thick sections were chipped and no cavity on the surface of the wire. Previous study that conducted by Narmada et al. [21] support this study by discovering that the surface morphology of NiTi archwire immersed at pH 4.2 was rougher than that of NiTi archwire immersed at pH 7.6. This study also consistent with the findings of Abalos et al. [13] who looked at NiTi archwire immersed in soft drinks with a low pH (pH 2.5) and then looked at the microstructure of...
the wire with a scanning electron microscope (SEM), found that the NiTi archwire's surface becomes rougher as the acidity rises [11].

NiTi archwire has a passive layer (TiO2) that serves as a corrosion barrier; however, if NiTi archwire is frequently in contact with solutions or foods with an acidic pH, the coating will be damaged over time. Damage to this passive layer reduces the wire's resistance to corrosion, allowing more ions to be released more easily in acidic pH conditions. The NiTi archwire soaked in tomato juice (Figure 2b) suffered more damage than the NiTi archwire soaked in orange juice (Figure 3b). The number of ions released in tomato juice was also higher than in orange juice (Table 1). This condition occurs because of the pH of tomato juice (4.7) is lower than orange juice (4.4).

Nickel is the metal that causes the most allergies. Oral mucosal cells from patients with fixed orthodontic appliances contained significantly more nickel than those from patients who did not have orthodontic appliances, according to previous study. According to previous study, nickel has a toxicological effect and has the potential to cause DNA cleavage in oral mucosal cells. The results of this study found that the highest average nickel ion released was 0.12 g, while the World Health Organization (WHO) recommends a daily dose of 25-35 g. Although this value is still significantly lower than the WHO-recommended daily dose, the number of released ions must still be considered in relation to the body's tolerance [23,24].

5. Conclusion
After immersion in tomato and orange juice, the number of nickel ions released and the surface microstructure of NiTi archwires differed significantly.

Acknowledgement
We thank the Faculty of Pharmacy University of North Sumatera, Physics Laboratory of Medan State University and Medan Environmental Health Engineering Center (BTKL) for providing facilities in this study.

References
[1] Cobourne M T and DiBiase A T 2016 Handbook of Orthodontics 2nd ed (Edinburgh: Elsevier) 1 311
[2] Cerroni S, Pasquantonio G, Condó R and Cerroni L 2018 Open Dent J 12 1 61
[3] Anusavice K J, Shen C, Rawls H R 2013 Phillips’ Science of Dental Materials 12th ed (Missouri: Elsevier) 40
[4] Proffit W R, Fields H W, Larson B E and Sarver D M 2019 Contemporary Orthodontics 6th ed (Philadelphia: Elsevier) 278
[5] Liu J K, Liu I H, Liu C, Chang C J, Kung K C, Liu Y T, et al 2014 Appl Surf Sci 317 6
[6] Ramazanzadeh B A, Ahhari F, Sabzevari B and Habibi S 2014 J Dent Res Dent Clin Dent Prospects 8 2 75
[7] Nonga H E, Simforian E A and Ndabikunze B K 2014 Tanzan J Health Res 16 4 1
[8] Rajauria G and Tiwari B K 2018 Thermal Pasteurization and Microbial Inactivation of Fruit Juices. In: Fruit Juices: Extraction, Composition, Quality and Analysis (London: Elsevier Inc) 317
[9] Gurnilang S A, Meidyawati R and Djauharie N 2018 J Phys Conf Ser 1073 3 1
[10] Heuvelink 2018 Tomatoes 2nd ed (Boston MA: CABl) 1
[11] Sharma M R, Mahato N, Cho M H, Chaturvedi T P and Singh M M 2018 Mater Technol 34 1 1 7
[12] Nanda R 2015 Esthetics and biomechanics in Orthodontics 2nd ed (Elsevier) 4
[13] Abalos C, Paul A, Mendoza A, Solano E, Palazon C and Gil F J 2013 J Mater Eng Perform 22 3 766
[14] Mikulewicz M, Wołowiec P, Loster B W and Chojnacka K 2015 J Trace Elem Med Biol 31 77
[17] Kristianingsih R, Joelijanto R and Praharani D 2014 J e-gigi 3
[18] Situmeang M A and Anindita P S 2016 Pharmacon 5 4 257
[19] Lubis H F, Harahap K I and Lubis D H 2020 Dent J (Majalah Kedokt Gigi) 53 2 69
[20] Hong M S, Kim S H, Im S Y and Kim J G 2016 Met Mater Int 22 4 628
[21] Lubis H F, Harahap K I and Lubis N T 2020 Maj Kedokt Gigi Indonesia 6 2 80
[22] William D and Callister D G 2015 Fundamentals of Materials Science and Engineering: An Integrated Approach 5th ed (Hoboken: Wiley) 714
[23] Narmada I B, Sudarno N T, Sjafei A and Setiyorini Y 2017 Dent J (Majalah Kedokt Gigi) 50 2 81
[24] Sianita P P and Iswari H 2011 Widya Maj Ilm 28 310 56
[25] Rasyid N I, Sri P and Heryumani J C 2014 Dent J (Maj Ked Gigi) 47 3 171