**RESEARCH ARTICLES**

**Nickel ion release of niti archwire in variations of immersion time and toothpaste**

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**ABSTRACT**

Nickel-titanium archwires in orthodontic wires have nickel ion releases. The use toothpaste and saliva immersion time may cause its releases. This study aims to prove the difference and interaction between immersion time and toothpaste variations towards the amount of nickel ion releases at nickel-titanium archwires. The study was an experimental research with a post-test only control group design. Twenty seven nickel-titanium archwires were divided into three groups, i.e., K group, which were immersed in artificial saliva. P1 was immersed in artificial saliva and 1.5 gram of toothpaste A, and P2 was immersed in artificial saliva, and 1.5 gram of toothpaste B. The samples were incubated (37 °C) for 1 and 1.5 months. Nickel ion released was analyzed using SSA. Two Way ANOVA and Post Hoc LSD test were the statistic test used. The results of this study were the K group which released 0.112 mg/l and 0.685 mg/l, P1 which released 0.093 mg/l and 0.670 mg/l, and P2 which released 0.099 mg/l and 0.657 mg/l. There were differences in toothpaste variations (p = 0.029), differences in immersion time (p = 0.000) and there was no interaction between immersion time and toothpaste variations (p = 0.505) on the amount of nickel ion release. There was a significant difference in K-P1 (p = 0.038), K-P2 (p = 0.013), P1-P2 (p = 0.049). There was a difference in immersion time and toothpaste variation, but there was no interaction between immersion time and toothpaste variation towards the amount of nickel ion releases.

**Keywords**: nickel ion releases; NiTi archwires; toothpaste variation; the length of immersion

**INTRODUCTION**

Orthodontic wire in braces would always have contact with saliva and mouth cavity tissue. The long placement of the Niti archwire in the mouth cavity could cause corrosion or the metal component’s release.¹²³ Metal components released from the Niti archwire could enter the body. It might carry local factors and cytotoxicity response in the body or regional influences or allergies in the form of the burnt sensation, hyperplasia gingiva, and faint at the side of the tongue.⁴ The emergence of allergies in the form of contact dermatitis was generally caused by nickel ion. The level of nickel ion increased in the saliva and serum significantly after the placement of orthodontic wires.² Nickel-titanium archwires are the frequently used material in orthodontic treatment. The archwires have nickel ion levels of more than 50%.⁵ The nickel ion contents can increase the hardness and resistance of orthodontic wires against corrosion. Corrosion is a metal reaction which will produce ion. It occurs when the metal surface directly contacts an environment containing oxygen and water.⁶ Some influencing factors are saliva pH, protein in the saliva, temperature, fluoride, and microorganism.⁷ Nickel-titanium archwires have the potential to have corrosion or ion release from the metal components in the mouth cavity. As in the previous research, nickel ion release occurred in nickel-titanium archwires immersed in artificial saliva with different immersion times, namely 1 hour, 24 hours, 1 week, 3 weeks, 1 month, 2 months.²⁵ The use of toothpaste could also cause corrosion. Toothpaste, in general, contains moisturizing materials, abrasive materials, anti-plaque materials, waters, fluorides, sweetener materials, flavors, binding materials, and detergents.⁸ The contents of some acids (such as citric acid and benzoate acid) and some sodium contents (such as sodium fluorides and sodium...
citric) are found in toothpaste and mouthwash. As in previous studies, nickel ion release occurs in nickel-titanium archwires immersed in various mouthwashes.9,10 Nowadays, toothpaste comes with various functions and tastes, such as orthodontic toothpaste, non-orthodontic toothpaste, and herbal toothpaste. Orthodontic toothpaste is used specifically for people who wear orthodontic wires. The composition contained in orthodontic toothpaste is similar to the standard toothpaste with colostrums11 as its additional composition. Detergent toothpaste is one of the commonly used non-orthodontic toothpastes. Sodium lauryl sulphates are the detergent materials contained in the toothpaste. Toothpaste with detergent could also cause the release of metal elements establishing alloy from the orthodontic wires since it has fluoride.12 By knowing this, dentists can be more aware of choosing toothpaste in patients who use fixed orthodontic appliances, with the clinical significance for the patients who have metal allergies with signs of burnt sensation, gingival hyperplasia, and faint at the side of the tongue.4 Based on the above background, researchers wanted to determine whether the length of immersion time and toothpaste variations could influence the number of ion releases of nickel-titanium archwires.

MATERIALS AND METHODS
This research was experimental laboratory research with a post-test only control group design. The samples were nickel-titanium orthodontic wires, a round shape with a diameter of 0.016 inches, a length of 2 cm, and a new wire. The total number of samples was 27 nickel-titanium archwires divided randomly into three groups, particularly 9 nickel-titanium archwires from the control group, 9 nickel-titanium archwires from treatment group 1, and 9 nickel-titanium archwires from treatment group 2. The control group was immersed in artificial saliva, the treatment group 1 was immersed in toothpaste A. The composition of the toothpaste A was aqua, hydrate silica, sorbitol sol, glycerin, steareth-30, carrageenan, aroma, titanium dioxide, disodium phosphate, 1500 ppm sodium fluoride, sodium benzoate, amyloglucosidase, citric acid, sodium saccharin, colostrum, allantoin, glucose oxidase, α-tocopheryl acetate, potassium thiocyanate, lysozyme, lactoferrin, lactoperoxidase; Treatment group 2 was immersed in toothpaste B. The composition of the toothpaste B was aqua, sorbitol hydrate silica, glycerin, 1.12% sodium monofluorophosphate, sodium saccharin, calcium carbonate, sodium lauryl sulphate (SLS), flavor, perlite, cellulose gum, sodium silicate, potassium citrate, DMDM hydantoin, O-phenylphenol, Cl 74160.

Nickel-titanium archwires (in the control group) were immersed in 20 ml of artificial saliva. In treatment group 1, nickel-titanium archwires were immersed in 20 ml of artificial saliva mixed with 1.5 grams of toothpaste A. In treatment group 2, nickel-titanium archwires were immersed in 20 ml of artificial saliva mixed with 1.5 grams of toothpaste B. The three treatment groups were incubated at 37 °C for one month. After one month, 10 ml of solution from each group was analyzed to identify the amount of nickel ion released. The rest of the available solution continued to be incubated at 37 °C for a half month, making the total duration of immersion and incubation of 1.5 months. After 1.5 months, 10 ml of solution from each group was analyzed to identify the amount of nickel ion released. The number of nickel ions released was examined using Atomic Absorption Spectrophotometry (Perkin Elmer Analyst 100) at the chemical laboratory of Universitas Negeri Semarang. The analytical test in this study used the two-way analysis of variance (ANOVA) test and continued with the post hoc LSD test.

RESULTS
Data from treated groups 1 and 2 showed fewer nickel ion releases than that of the control group (Table 1). The data of the number of NiTi archwires
Table 1. Descriptive analysis of the number of Nickel Ion Releases of NiTi Archwires (mg/l) after being immersed for 1 month and 1.5 months

<table>
<thead>
<tr>
<th>Group</th>
<th>Immersion time</th>
<th>1 month</th>
<th>1.5 months</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td></td>
<td>0.112 ± 0.027</td>
<td>0.685 ± 0.013</td>
</tr>
<tr>
<td>Treated Group 1</td>
<td></td>
<td>0.093 ± 0.025</td>
<td>0.670 ± 0.015</td>
</tr>
<tr>
<td>Treated Group 2</td>
<td></td>
<td>0.099 ± 0.017</td>
<td>0.657 ± 0.040</td>
</tr>
</tbody>
</table>

Information:
Control Group: titanium nickel archwires immersed in artificial saliva
Treated Group 1: titanium nickel archwires immersed in artificial saliva and toothpaste A
Treated Group 2: titanium nickel archwires immersed in artificial saliva and toothpaste B

Figure 1. The graph of average number of releases of control group, treated group 1 and treated group 2 of ion nickel after being immersed for 1 month and 1.5 months

Table 3. The result of two-way anova test

<table>
<thead>
<tr>
<th></th>
<th>Value of p§</th>
</tr>
</thead>
<tbody>
<tr>
<td>Treatment</td>
<td>0.029*</td>
</tr>
<tr>
<td>Length of immersion time</td>
<td>0.000*</td>
</tr>
<tr>
<td>Treatment length of immersion time</td>
<td>0.505</td>
</tr>
</tbody>
</table>

Information: *means (p < 0.05); §Two-Way Anova

Nickel ion releases immersed for one month in Table 1 showed normal data distribution (p > 0.05), and it was homogeneous with the significance value of 0.089 (p > 0.05).
Data of treated groups 1 and 2 had less nickel ion release than that of the control group. The comparison of the amount of nickel ion releases from nickel-titanium archwires in the control group, treated group 1, and treated group 1 is described in the following figure (Figure 1).

The three groups average number of nickel ion release still increased after being immersed for 1 month and continued for another 1/2 month. On the 1 first month of immersion, treated group 1 had fewer nickel ion releases than the treated group 2. Still, after 1.5 months of immersion, the number of nickel ion releases from treated group 1 increased and was higher than the treated group 2 (Figure 1).

The results of the Two-Way ANOVA test showed that the amount of nickel ion releases of nickel-titanium archwires after immersion in the control, treatment 1, and treatment 2 groups showed significant results with a p-value of 0.029 (p < 0.05) (Table 3).

The number of nickel ion releases of nickel-titanium archwires showed a significant result with a p-value of 0.000 (p < 0.05) in each treated group compared between 1 month immersion time and 1.5 months immersion time (Table 3). After 1.5 months of immersion, treated group 2 had a decrease in nickel ion releases compared to the treated group 1 (Figure 1). Furthermore, an interaction analysis was carried out in the immersion time and the type of toothpaste to see some activities that influencing each other. The two-way ANOVA test results towards the length of immersion times and a treated group of toothpaste variations showed no interaction with p-value of 0.505 (p > 0.05). On this basis, there is no difference in the amount of nickel ion release in terms of the combination between immersion time and variations of toothpaste.

The post hoc test results showed a significant difference in the three pairs of treated groups in this research. Table 4 presents a significant difference between treated group 1 and treated group 2 with a p-value of 0.049. It means that there were different numbers of nickel ion releases from nickel-titanium archwires after being immersed in toothpaste A and toothpaste B.

DISCUSSION

Nickel-titanium archwires used in orthodontic treatment might have nickel ion releases since it is constantly in contact with the environment within the mouth cavity during the treatment period, such as temperature, microflora, and the degree of acidity or saliva pH. The nickel-titanium archwires’ releases of nickel ion were also affected by food or beverages consumed and the dental treatment products used such as toothpaste, mouthwash, and topical fluoride.8,12,13

The average number of nickel ion releases on nickel-titanium archwires in each group and period of immersion could be affected by the solutions’ pH. The pH of solutions in the treated group 1 after mixing 1.5 gram of toothpaste A into 20 ml artificial saliva was 7.2, pH of solutions in the treated group 2 after mixing 1.5 gram of toothpaste B into 20 ml artificial saliva was 7.4, while the pH of artificial saliva of the control

<table>
<thead>
<tr>
<th></th>
<th>Control</th>
<th>Treated group 1</th>
<th>Treated group 2</th>
</tr>
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<tbody>
<tr>
<td>Control</td>
<td></td>
<td>0.038*</td>
<td>0.013*</td>
</tr>
<tr>
<td>Treated group 1</td>
<td>0.038*</td>
<td></td>
<td>0.049*</td>
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<tr>
<td>Treated group 2</td>
<td>0.013*</td>
<td>0.049*</td>
<td></td>
</tr>
</tbody>
</table>

Information: * means (p < 0.05)
Control group: titanium nickel archwires immersed in artificial saliva
Treated group 1: titanium nickel archwires immersed in artificial saliva and toothpaste A
Treated group 2: titanium nickel archwires immersed in artificial saliva and toothpaste B
group was 7. The average amount of nickel ion releases in the control group was higher than that of the treatment group 1 and treatment group 2. The lower level of pH can cause this in the two treatment groups. This result is in line with the research by Hazazi suggesting that there were nickel ion releases of nickel-titanium archwires after being immersed in artificial saliva with normal pH for 1, 14, 21, and 28 days. Artificial saliva had several substance components, one of which was chloride ion that could cause nickel ion releases through electrochemistry reaction. It occurred in the oxidized anode and reduced cathode. Artificial saliva mixed with toothpaste would increase the pH of the saliva. It was because the fluorides contained in the toothpaste, besides, the detergent contents such as sodium lauryl sulphates in toothpaste also supported the increase of saliva pH. The rise of saliva pH could also influence the number of nickel ion releases of nickel-titanium archwires. We can see in table 2 and table 3 that the number of nickel ion releases on the treated group 1 and 2 were lower than that of the control group.

The results showed that there was a significant difference in the amount of nickel ion releases of nickel-titanium archwires immersed in the control group, treatment 1, and treatment 2. There was a significant difference in the amount of nickel ion releasing nickel-titanium archwires in this study between the control group, treatment 1 and treatment 2. This result could be attributed to the pH of the solutions. The Control group consisted of artificial saliva with pH 7, treated group 1 consisted of artificial saliva with pH 7, which after being mixed with toothpaste A, the pH became 7.2. Meanwhile, treated group 2, which consisted of artificial saliva and mixed with toothpaste B, had pH 7.4. According to Raymond et al., toothpaste consisting of sodium lauryl sulphates had pH 7.4 - 9.5. pH of toothpaste A, and toothpaste B solutions could sufficiently cause corrosion or metal component releases such as nickel ion on nickel-titanium archwires. Oxygen reduction reaction on the archwire’s surface produced OH- ion that could increase pH value. Oxidized iron would settle forming a deposit, so that it caused corrosion or metal component releases.

Fluorides could also cause nickel ion releases from nickel-titanium archwires. Titanium oxide layers covering nickel-titanium archwires would be broken if fluorides interact with those layers and become sodium hexafluorotitanate. Titanium oxide layers were the protective layers added to the nickel-titanium archwires to prevent corrosion or release one of the metal components. Fluorides contained in toothpaste A in this research were 1500 ppm, while toothpaste B had 1.12%. The number of fluorides had been able to cause nickel ion releases from the nickel-titanium archwires.

Metal component releases such as nickel ion on nickel-titanium archwires could be caused by acid contents in toothpaste A such as disodium phosphates, sodium fluorides, citric acids, sodium saccharins, and in toothpaste B such as sodium lauryl sulphates, sodium monofluorophosphates, sodium silicates, sodium saccharins, sodium laureth sulphates. It was because the environment and the solution components for immersion in the nickel-titanium archwires could affect the level of corrosion on the wires. The research by Minanga et al. suggested that there had been nickel ion releases and chromium on the stainless steel archwires after being immersed in the mouthwash with acid contents such as citric acids, benzoic acids, and several types of sodium, such as sodium fluorides, sodium citrates, sodium benzoates, sodium lauryl sulphates, and sodium saccharine. Citric acids had sufficiently high H+ particles that could increase if it interacted with metal which could also increase corrosion. The temperature used in this research was 37 °C. Temperature also affected the characteristics and the number of nickel ion releases of the nickel-titanium archwires. The higher the temperature, the higher the ion releases. It was because the kinetic energy from the particles reacted would increase beyond the value of
activation energy and increased the reaction velocity rate (corrosion). Temperature of 37 °C had already been able to release nickel ion.\textsuperscript{21,22}

The number of nickel ion releases after 1 month of immersion compared to the 1.5 months of immersion in each group showed a significant difference. Nickel ion releases in the control group, treated group 1, and treated group 2 with 1 month immersion and continued until the total 1.5 immersion could not increase the number of nickel ion released (table 2). This result indicated that the immersion time also affected the number of nickel ion releases from the nickel-titanium archwires. The number of nickel ions released in this research still had increased up to 1.5 months, which could be affected by pH and the temperature in this research. The number of nickel ion released in the treated group 1 in 1 month immersion was less than in the treated group 2, which was attributed to the fact that toothpaste A did not contain detergents, so that the pH of solutions after being mixed with 1.5 gram of toothpaste A only increased a bit from the normal pH. Released nickel ion in the treated group 2 in 1.5 months of immersion was less than the treated group 1. It could be caused by the decreasing saliva pH for long term usage of the toothpaste with detergents.\textsuperscript{23}

The result of Two-way Anova test showed that there was no interaction between the length of immersion time and toothpaste variations. It could be attributed to the fact that the different number of nickel ion of NiTi archwires on the treated group was not overly high. In addition, it could be caused by the composition of toothpaste, such as fluorides and alkaline pH on the soaking solutions.\textsuperscript{15,17,24} The post hoc test result showed a significant difference between the control group and treated group 1, control group with treated group 2 and treated group 1 and treated group 2. It could be resulted from the pH of each group, the number of fluorides in treated group 1 and treated group 2, and the temperature used in this research.\textsuperscript{3,19,21,22}

The limitation in this research was the fact that the toothpaste only used the ones available in the market, and thus the researchers could not control the composition and the concentration in the toothpaste.

CONCLUSION
Orthodontic toothpastes and detergent toothpastes can cause significant release of nickel ions from nickel-titanium archwires after immersion for 1 month and 1.5 months. There was no interaction between immersion time and toothpaste variations on the amount of nickel ion releases of nickel-titanium archwires.

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