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THE DIFFERENCE OF SURFACE MORPHOLOGY AND CHEMICAL COMPOSITION OF SUPER-ELASTIC NICKEL TITANIUM ORTHODONTIC WIRES BEFORE AND AFTER IN ARTIFICIAL SALIVA SUBMERSION

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ABSTRACT

Objective: *In order to Investigate the difference of morphology and composition of Super-elastic Nickel Titanium wire before and after submersed in artificial saliva with normal pH (pH 6:50) and acidic pH (pH 3.75).* ***Material and method:*** *Super-elastic Nickel Titanium wire were divided into 2 groups. Each group consisted of 3 samples submerged in 150 ml of artificial saliva for 28 days. The first group was Super-elastic Nickel Titanium wire submersed in artificial saliva with normal pH (pH 6.50) while the second group was submersed in artificial saliva with acidic pH (pH 3.75). Wire surface morphology was Analysed using Scanning Electron Microscope (SEM) while the wire composition was Analysed using Energy Dispersive X-ray Spectroscopy (EDS.* ***Result:*** *Scanning Electron Microscope revealed a significant difference of surface morphology of super-elastic nickel titanium before and after submersed in artificial saliva between normal pH (pH 6.0) and acidic pH (pH 3.75). SEM investigation Showed a fibrous microstructure on the surface of super-elastic nickel titanium before submersed in artificial saliva and became rough and showed many form of pitting or crevice corrosion after submersed in artificial saliva for 28 days. The chemical compositions in surface morphology were analysed using EDS Also showing a different result. The major compositions of super-elastic nickel titanium wire were the C, N, Ni and Ti and after submersed in artificial saliva for 28 days has made any additional elements O, Al, Si, P, Cl, Ca, K and Fe.* ***Conclusion:*** *the surface morphology of nickel titanium surface rugosity greater SEM Showed and presence of many forms of pitting or crevice corrosion after submersed in artificial saliva with normal pH and acidic pH. The major surface composition of a super-elastic nickel titanium before submersed in artificial saliva were the C, N, Ni and Ti and as a major composition of super-elastic nickel titanium before and after submersed in artificial saliva with normal pH (pH 6.0) and acidic pH (pH 3.75).*

Keywords: *Orthodontic Wire, Super-elastic Nickel Titanium, surface morphology, chemical composition*

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INTRODUCTION

Wire types that are often used to move the orthodontic tooth is a wire made of Stainless Steel, b-titanium, Cobalt Chromium-Nickel or Nickel Titanium. Nickel Titanium was first used in the orthodontics field in 1970 due to their superior properties are the mechanical and chemical properties. Nickel Titanium wire becomes very popular because it is elastic, 20% more elastic (super-elastic), thermal shape memory and demonstrate the ability resistant to corrosion and biocompatible. The use of Nickel Titanium wire makes the orthodontic treatment more efficient and influence the overall treatment.[1,2] The combination

of all materials used in the orthodontic treatment works under pressure in intra oral area. These pressures include saliva, other liquids, temperature fluctuations, mastication and load the application itself. This combination can cause corrosion reaction, the degradation process of a metal and the release of metal ions into the fluid as a result of an electrochemical reaction which will further degrade the mechanical properties of the metal.[1,3] The resistance of a wire is an important aspect of biocompatibility can be affected by several factors. The first factor is influenced by the manufacturing process, the type of metal and surface characteristics (morphology), the second factor is influenced by the environment and the third factor is influenced by how long the metal used (aging), which depends on the pressure, temperature treatment, and recycling processes.[4] The purpose of this study is to determine differences in morphology and composition of the chemical element Super-elastic Nickel Titanium wire before and after artificial saliva soaked with pH 6,50 and pH 3,75.

MATERIALS AND RESEARCH METHODS

Materials

This research was conducted at the Laboratory of Chemistry, Faculty of Pharmacy, University of Muslim Indonesia is to produce artificial saliva; Microbiology Laboratory of the Indonesian Muslim University for the submersion in an incubator anaerobic; and laboratory BATAN (National Nuclear Energy Agency) in Jakarta for analysis of SEM / EDS (Scanning Electron microscope / Energy Dispersive X-Ray Analyzer JED-2300 series, JEOL Ltd. Tokyo, Japan)

The sample for this study is the Super-elastic Nickel Titanium wire with a round cross-sectional shape, size 0.016 inch, ovoid curved shape, length 12 cm and from the same manufacturer: *Orthomax*. Total samples for initial morphology test and chemical composition test before submersing the elements of an artificial saliva are 3 pieces. The number of samples to test morphology and chemical composition test element after artificial saliva submersion for each group as well as 3 pieces of samples. The treatment group was divided into two groups, group with immersed in artificial saliva pH 6,50 and in pH 3,75 was 3 pieces.

Surface Morphology Test

It is aimed to know the surface morphology Super-elastic Nickel Titanium wire before and after artificial saliva soaked with 2 kinds of different pH: 6,50 and 3,75. This test is performed by using a Scanning Electron Microscope (SEM) / Energy Dispersive X-Ray Spectroscopy (EDS) (Scanning Electron microscope / Energy Dispersive X-Ray Analyzer JED-2300 series, JEOL Ltd. Tokyo, Japan).

Surface Composition Test

The wire surface composition test is performed using an Energy Dispersive X-ray Spectroscopy (EDS), it aims to determine the type and percentage of component elements making up the wire. This test is conducted to determine the type and percentage of constituent components of the wire before and after artificial saliva soaked with pH 6,50 and pH 3,75.

RESEARCH RESULT

Surface Morphology Test Results Using *Scanning Electron Microscope*

The wire used in this study is the kind of Super-elastic Nickel Titanium belonging to the prefabricated wrought alloy. The wrought alloy is a metal alloy that is formed or produced by forging. The microstructure of a metal alloy forged with their creation is fibrous.[5] This is consistent with observations on the surface of the Super-elastic Nickel Titanium wire which is shown in Figure 1. It shows the surface morphology of Super-elastic Nickel Titanium wire before soaked artificial saliva has fibrous surfaces, the wire surface was rough enough, found some scratch and several cavities. This fibrous microstructure indicates the results of the cold working process manufacturers, in according to the theory that the cold working is the process of making the metal below the solidus temperature that causes fibrous microstructure in a metal alloy.[5] The cold work on a wire production process can trigger the initial deformation of the wire, which was marked by irregularities in the surface.[6]

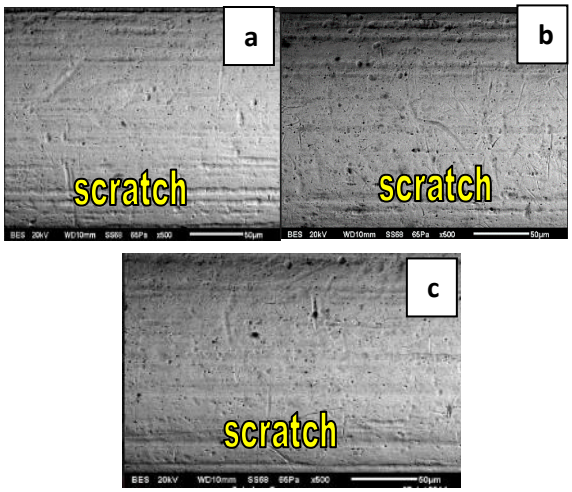

Figure 1. The test results from Super-elastic Nickel Titanium wire surface morphology that use SEM before soaked artificial saliva.

The surface observation result of Super-elastic Nickel Titanium after soaking the artificial saliva with pH 3,75 and pH 6,50 shown in Figure. 2 shows the effects of submersion in artificial saliva which triggers the degradation of the surface of the wire (releasing of wire constituent elements) which is characterized by the wire surface becomes rougher with some bigger cavities . This indicates there has been a presence of pitting corrosion on the surface of the Super-elastic Nickel Titanium wire after being soaked with artificial saliva. Figure 2 (a), (b) and (c) shows the surface of the Super-elastic Nickel Titanium wire after artificial saliva soaked with a pH of 3,75, it appears the pitting corrosion measuring $\pm$ 20 µm, while pitting corrosion that occurs on the surface of the Super-elastic Nickel Titanium wire soaked artificial saliva with a pH of 6,50 indicates a larger size with size $\pm$ 40 µm.

The surface of a metal is a major factor of resistance of metal against corrosion if there is a scratch and any irregularities in the surface of the metal are experiencing a cause corrosion.[3] This happened because of the initial deformation on the wire, causing irregularities in the surface, facilitate a solution of saliva into the defect and will trigger the process of corrosion and increases with exponentially with time.[7] Corrosion tends to occur in areas that have suffered deformation. The rough part usually has lost the protective film (anode) so that electrochemical reaction occurs with a smooth part (cathode) with saliva. This reaction corrode.[8] Other studies that agree it is also said that the defects on the surface of Nickel Titanium wire generated during the production process can accelerate corrosion.[7]

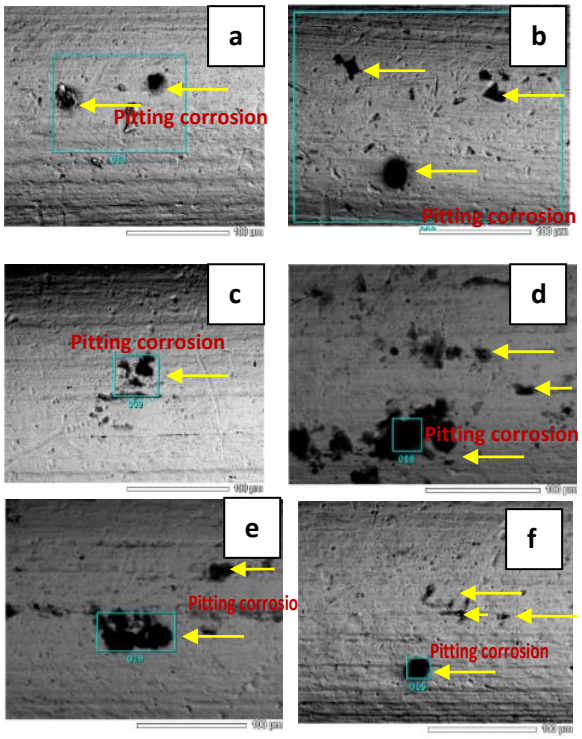

Figure 2. The SEM results of Super-elastic Nickel Titanium wire surface after soaked in artificial saliva. (a) (b) (c) pH 3,75 and (d) (e) (f) pH 6,50 (500x magnification)

The Results of Wire Chemical Elements Composition Using Energy Dispersive X-ray Spectroscopy (EDS)

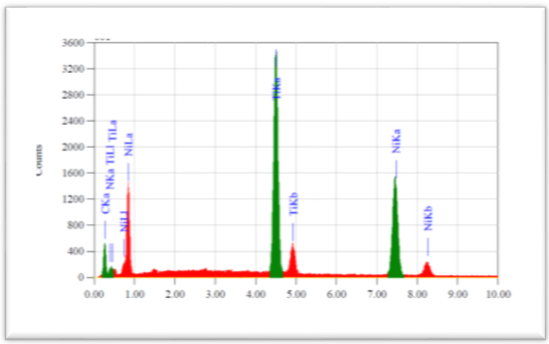

Figure 3. EDS pattern of Nickel Titanium SE before soaked artificial saliva.

The results of the chemical composition testing using EDS aims to analyse chemical elements on the wire surface. The elements of wire Super-elastic Nickel Titanium before soaked in artificial saliva are C, N, Ti, Ni (figure 3). The comparison of each element in the wire, Super-elastic Nickel Titanium are shown in Table 1.

Table 1. Weight percentage (wt%) by EDS analysis before soaked in artificial saliva.

Element	Mass (Wt%)			Average
	A	B	C	
C	13.21	12.87	13.01	13.03
N	5.47	3.55	6.15	5.06
Ti	35.34	36.24	34.67	35.42
Ni	45.98	47.34	46.18	46.50

It is seen that the main component of this wire is Nickel with an average weight percentage of 46,5 Wt% and Titanium 35,42 Wt% so that the alloy of metal is called a metal-based Nickel-Titanium (Nickel Titanium-based alloy). Other metal elements as the elements carbon and nitrogen are added to improve the performance of the Super-elastic Nickel Titanium wire such as changes in hysteresis properties, temperature change and an increase in the ability of mechanical properties. At first, the alloy of metal and large composition of Nickel were avoided by clinicians due to the release of Ni to the corrosive environments. The human body fluid prefers the alloy of metal and small composition of Nickel such as Stainless steel, Titanium, and Titanium with Cobalt based metal alloy. The atomic bond between Nickel and Titanium is a very strong bond that will be difficult for the atoms in it to leave the bonding. But element Titanium easily oxidized, thus forming the oxide layer as a protective coating to resist diffusion and release of Nickel element which led both metal alloy led to corrosion resistance.[6]

The effects after soaking the Super-elastic Nickel Titanium wire in artificial saliva with pH 3,75 and pH 6,50 shows the difference in the elemental composition of the surface of the wire are significant (Table 2). There are some new elements such as C, O, K, P, Fe, Al, Cl and Ca, in addition to the elements C, N, Ti and Ni that are main constituent elements of the Super-elastic Nickel Titanium wire. Based on the results we can conclude that those new elements are the constituent elements of artificial saliva that undergoes a chemical reaction with the surface of the Super-elastic Nickel Titanium wire. The similar result to previous studies on the wire surface is that there is deposits derived from saliva, they are the elements of Na, K, and Cl in a small number of atoms, and elements of Ca and P in the lots of numbers.[10]

Table 2. Weight percentage (wt%) by EDS analysis after submersion of artificial Saliva with pH 3,75 and pH. 6,50

Element	Average Weight (Wt%)	Average Weight (Wt%)
	pH 3.75	pH 6.50
C	13.94	24.33
N	4.74	2.71
O	6.03	14.82
Al	0.72	0.10
Si	0.11	0.83
P	0.20	3.45
Cl	0.3	0.55
Ca	32.20	4.25
Ti	41.25	20.85
Ni	0.15	27.17
K	0.21	0.43
Fe	0.37	0.71

The results of EDS on the Super-elastic Nickel Titanium wire soaked artificial saliva with pH 6,50 found any element of O with a considerable amount which is the average percentage of 14,82 Wt%. The O element is the result of surface passivation on wire bonded with Ti element forming an oxide layer of TiO2. The weight different of Ti element can be seen in the research that the amount of Ti element was reduced from 35,42% Wt to 20,85Wt% Wt, because of the binding with O elements that form the passivation layer. The number of main constituent elements of the Super-elastic Nickel Titanium wire (Ni, C and N) also decreased due to the degradation process.

The Super-elastic Nickel Titanium submersion in artificial saliva with an acidic pH (pH 3,75) result some new elements in addition to the main elements making up the wire they are C, N, Ti, and Ni. The percentage by weight of each of the elements after soaking shown in Table 2. Changes also occur in the constituent elements of the Super-elastic Nickel Titanium wire after the submersion of artificial saliva with a pH of 3,75. In the submersion with a pH of 3,75, the elements Ni and Ti as the main constituents of the wire element is only slightly decreased the amount, for the element Ni from 46,50 Wt% to 41,25 Wt%, while

the element Ti from 35.42% to 32.20 Wt% Wt. Elements C and N did not undergo significant changes in artificial saliva submersion with acidic pH (pH 3,75). Element C only slightly increased from 13,03 Wt% to 13,94 Wt%, while the N element decreased slightly from 5,06 Wt% to 4,74 Wt%.

The submersion results of Super-elastic Nickel Titanium wire at a pH of 3.75 was also seen their saliva deposits attached to the second surface of the wire. These elements are Cl, K, P, Ca, Al, Si, Mg and O. Element O on submersion with a pH of 3.75 was found with less percentage of only 6,03 Wt% when compared to the O elements found in the wire submersion pH 6.50 with a considerable amount of O that has 14,82 Wt%.

In Table 2 shows a Cl element of the Super-elastic Nickel Titanium wire a different percentage. Cl amount looks larger on the Super-elastic Nickel Titanium wire submersed in artificial saliva pH 6,50 (0,55 Wt%) compared with the wire immersed in artificial saliva pH 3,75 (0,3 Wt%). If it is connected to the percentage of Ni elements which has been degraded, it appears that on the Super-elastic Nickel Titanium wire submersed artificial saliva with a pH of 6,50, the percentage of Ni ion has greater degradation than the wire submersed in artificial saliva with a pH of 3,75. Cl element binds with water and produces acid which in turn will trigger the release of the Ni element as one of the initial processes of corrosion.[11] Artificial Saliva has the effect of corrosion due to the element Cl. Cl element causes pitting corrosion.[12] This result is according to the results of this study to found more Cl element in the Super-elastic Nickel Titanium wire submersed with artificial saliva pH 6,50 and has a larger pitting corrosion. The Super-elastic Nickel Titanium wire submersed in artificial saliva with pH 6,50 had more amount in releasing of the wire elements compared with the release of Super-elastic Nickel Titanium wire elements submersed with pH 3,75 causes the number of cavities pitting corrosion more (seen in the SEM Figure 2).

CONCLUSION

There were morphology changes on the surface of Super-elastic Nickel Titanium wire after submersed to artificial saliva at pH 6,50 and pH 3,75; it was the formation of pitting corrosion on the surface of the super-elastic Nickel Titanium wire. In the composition of chemical elements changed after submersing to the artificial saliva at pH 6,50 and pH 3,75; besides there are new elements as the result of chemical reactions between artificial saliva and Super-elastic Nickel Titanium wire.

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