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Source / Izvornik: Dental Materials Journal, 2017, 36, 149 - 156

Journal article, Published version
Rad u časopisu, Objavljena verzija rada (izdavačev PDF)

https://doi.org/10.4012/dmj.2016-169

Permanent link / Trajna poveznica: https://urn.nsk.hr/urn:nbn:hr:184:767847

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Download date / Datum preuzimanja: 2021-01-23

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Effect of pH, fluoride and hydrofluoric acid concentration on ion release from NiTi wires with various coatings

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Aim was to determine effect of pH, fluoride (F−) and hydrofluoric acid concentration (HF) on dynamic of nickel (Ni2+) and titanium (Ti4+) ions release. Nickel-titanium wires with untreated surface (NiTi), rhodium (RhNiTi) and nitride (NNiTi) coating were immersed once a week for five min in remineralizing agents, followed by immersion to artificial saliva. Ion release was recorded after 3, 7, 14, 21 and 28 days. Pearson correlations and linear regression were used for statistical analysis. Release of Ni2+ from NiTi and NNiTi wires correlated highly linearly positively with HF (r=0.948 and 0.940, respectively); for RhNiTi the correlation was lower and negative (r=−0.605; p<0.05). The prediction of Ti4+ release was significant for NiTi (r=0.797) and NNiTi (r=0.788; p<0.05) wire. Association with F− was lower; for pH it was not significant. HF predicts the release of ions from the NiTi wires better than the pH and F− of the prophylactic agents.

Keywords: Topical fluoride, Mass spectrometry, Nickel-titanium alloy, Orthodontic wires, Surface properties

INTRODUCTION

Various modifications of the surface of NiTi archwires are being introduced in attempt to keep wires’ excellent working properties while improving their aesthetic appearance and reducing susceptibility to corrosion1,2. Nitrogen ion implantation leads to surface composition changes from NiTi to TiN (titanium nitride), replacing known allergen nickel with inert nitrogen. Improvement of the corrosion resistance of NiTi wire with nitrified surface compared to the uncoated NiTi wire was confirmed in previous electrochemical researches performed in artificial saliva and fluoride mouth rinse solution3,4. Rhodium is a noble metal with white reflective appearance and excellent anti-corrosion properties5, but in the commercially available wires it was noted that this aesthetic feature diminishes fast within oral cavity6,7. Also, it was found that this surface coating was uneven and non-homogeneous, which caused susceptibility to corrosion in artificial saliva, due to formation of galvanic coupling of underlying NiTi to noble metals in the surface coating8-10. There are no data available regarding the interaction between the rhodium coating and fluoride solutions.

Design of fixed orthodontic appliance enables retention of plaque which reflects in rapid change in microbial composition; with increase in acidogenic Streptococcus and Lactobacilles species8,9. Lactic acid is the metabolic product that keeps the pH of the plaque low (in 24 h plaque pH is 4.8)8,10; also, during longer inactive periods (e.g. sleeping) the pH of saliva is below 5, as well as after food intake (increase in bacterial metabolism also causes drop of the pH of saliva below 5)11,12.

Prophylactic agents are being used as adjuvant therapy during an orthodontic treatment to combat increased incidence of white spot lesions13. Adjuvant fluorides and calcium are being used for surface and subsurface remineralization14, and repeated administration of adjuvant prophylactic agents is recommended and necessary for maintenance of intact tooth surface in conditions of challenged oral hygiene15. Fluorides interact with the nickel-titanium (NiTi) orthodontic archwires, where protective titanium-oxide (TiO2) layer on surface of the wires forms complexes with fluorides, and leads to surface corrosion, releasing nickel (Ni2+) and titanium (Ti4+) into adjacent tissue and saliva16. The metal forming more stable corrosion products corrodes slower; therefore the Ni2+ ions are being released into the environment more than the Ti4+ ions17.

Previous research recorded the highest Ni2+ ion release from fixed orthodontic appliances during the first week of immersion in artificial saliva, and significant retardation in the Ni2+ ion release with prolonged immersion time18,19. The titanium tends to form passive oxide layer on the surface, it was not established whether repeated application of fluoride containing agents reduces capability to restore TiO2 protective layer on the NiTi wires, and affects the wire’s corrosion susceptibility. Furthermore, if the protective ceramic or metallic coatings are non-homogeneous or contain micro cracks, there is a potential for formation of the localised corrosion within the underlying alloy due to
the invasion of the corrosive agent\textsuperscript{[28]}.

Damaged surface layers increase the nickel ion release from the NiTi wire and consequently reduce the wire’s biocompatibility, which can manifest as the hypertrophic gingival tissue and hypersensitivity reactions\textsuperscript{[16,27]}.

The fluoride concentration (F\textsuperscript{−}) and the pH of the solution influence the corrosion susceptibility of the NiTi wires\textsuperscript{[12,22,23]}, where the acidic fluoride environment favours the formation of the hydrofluoric acid (HF) which destroys the TiO\textsubscript{2}\textsuperscript{[24]}. The influence of regular weekly application of remineralizing agents on wire’s corrosion resistance and the dynamic of the release of ions from NiTi wires have not been established. Also, the influence of the type of coating on ion release from NiTi wires during regular use of remineralizing agents was not determined. Commercially available fluoride containing agents have many adjuvant ingredients, in range from phosphoric acid (used to decrease the pH of the agent) to various aromas (to improve flavour). Because of all this co-founders, this research tried to single out the most important contributors, which are common to various prophylactic re-mineralizing agents. The data on F\textsuperscript{−} and pH are easily available from the data sheet for every commercial prophylactic re-mineralizing agent, while the HF can be calculated from the F\textsuperscript{−} and pH. Therefore, this study aims to determine effect of pH, F\textsuperscript{−} and HF concentration on dynamic of the release of nickel (Ni\textsuperscript{2+}) and titanium (Ti\textsuperscript{4+}) ions from NiTi wires with various coatings induced by repeated weekly use of the fluoridated remineralizing agents.

\section*{MATERIALS AND METHODS}

\textbf{Materials}

Preformed NiTi orthodontic archwires in dimensions 0.508×0.508 mm BioForce Sentalloy (Dentsply GAC, NY, USA) were used in this study. The wire types used were: 1) NiTi with untreated surface; 2) rhodium coated NiTi (High Aesthetic); 3) nitride coated NiTi (IonGuard).

Their chemical composition (titanium 49–50 wt%, nickel 50–51 wt%) was determined with the help of the atomic absorption spectrometry on Atomic Absorption Spectrophotometer, type AA 6800 (Shimadzu, Kyoto, Japan). The wire specimens for the AAS were dissolved from standard solutions (1,000 mg/L (Ti); 500 mg/L (Ni); 250 mg/L (F)) and kept in a heated water bath at 37°C. Average F\textsuperscript{−} and pH values were: Mirafluor-k-gel 5.1; Elmex gelée 5.5; MI Paste Plus 6.6. The F\textsuperscript{−} was measured with fluoride-ion selective electrode on Expandable Ion Analyzer EA 940 (Orion Research, Beverly, MA, USA). Average F\textsuperscript{−} was: 1) Elmex gelée 11273.3 ppm; 2) Mirafluor-k-gel 6171.1 ppm; 3) MI Paste Plus 816.4 ppm. The HF was calculated from the F\textsuperscript{−}, the pH of the remineralizing agents and the HF acid dissociation constant (pK\textsubscript{a}=3.17) according to the equation: [HF]=([F\textsuperscript{−}]/[10\textsuperscript{pK\textsubscript{a}}−\textsuperscript{pH}]). The HF used for further analysis were: 1) MI Paste Plus 0.32 ppm; 2) Elmex gelée 53.88 ppm; 3) Mirafluor 71.31 ppm.

\textbf{Methods}

The wire specimens, each 10 cm long and 2 cm² in surface, were immersed in 10 mL of artificial saliva and kept in a heated water bath at 37°C. At one week intervals, the wires were taken out of the artificial saliva, and immersed in MI Paste Plus, Mirafluor-k-gel and Elmex gelée for five min at 37°C. Afterwards, the wire specimens were rinsed with distilled water and immersed to fresh artificial saliva. The Ni\textsuperscript{2+} and Ti\textsuperscript{4+} ion release was recorded after 3, 7, 14, 21 and 28 days of immersion. Released Ni\textsuperscript{2+} and Ti\textsuperscript{4+} ions were measured by inductively coupled plasma-optical emission spectroscopy on Thermo Elemental “IRIS Intrepid II” XSP, Duo, (Thermo Electron, Austin, TX, USA). The average daily nickel release during the observed period was calculated for two wires (for upper and lower dental arch), in order to translate the results to clinical setting and address the aspect of biocompatibility\textsuperscript{[30]}. All experiments were done in triplicate.

Surface characterization was employed to demonstrate and help clarify corrosion processes, as they primarily occur on the wire’s surface. Upon completion of the ion release immersion protocol, one wire from every experimental group was taken for surface analysis to record surface changes related to ion release. Visual inspection of the surface was done with the scanning electron microscopy (SEM) FEG QUANTA 250 (FEI Company, Eindhoven, The Netherlands) at magnification ×2,000. The analysis of the chemical composition of the surface and near surface areas (four different spots on one wire from every experimental group, both bright and dark areas) was done with the use of energy-dispersive spectroscopy (EDS) QUANTAX EDS (Bruker, Karlsruhe, Germany).

\textbf{Statistical methods}

The sample size was based on a priori power analysis. For the expected correlation coefficient r=0.75, the statistical power δ=80% and significance level α=5%.
the needed minimum sample size for testing of correlations and the regression analysis describing ion release influenced by the HF concentration equals 11, for every wire type (MedCalc 14.8.1, MedCalc Software, Ostend, Belgium). Pearson correlation and linear regression were used to explore influence of the pH value, total fluoride concentration and HF concentration in prophylactic agents on the ion release from the orthodontic wires. Effect sizes i.e. the magnitude of relationship was estimated by using r and $R^2$. The SPSS 10.0 (SPSS, Chicago, IL, USA) was used for all analysis, statistical significance was pre-set to $p<0.05$.

Previously published results from wires’ immersion to the artificial saliva alone were used for comparison.

**RESULTS**

**Release of Ni$^{2+}$ and Ti$^{4+}$ ions**

The release of Ni$^{2+}$ and Ti$^{4+}$ ions from the three wire types combined with various remineralizing agents is shown in columns (for measured time intervals), and as lines (total amount) over time in Fig. 1. The NiTi and NNiTi wires showed similar behaviour in remineralizing agents, with most of the ions released after immersion to the media with the highest HF concentration. The RhNiTi wire released most of the ions in combination with the media with the lowest HF concentration.

**Correlation between the pH, $F^-$ and HF concentration in remineralizing agents and the release of Ni$^{2+}$ and Ti$^{4+}$ ions**

Table 1 shows the correlations between the release of the Ni$^{2+}$ and Ti$^{4+}$ ions from the tested wires and the pH value, $F^-$ and HF in remineralizing agents determined with the Pearsons correlations. The HF from the remineralizing agents exhibited the highest correlation with the release of the ions from the NiTi wires. The release of the Ni$^{2+}$ ions from the NiTi and NNiTi wires increased with the increase in the HF of the remineralizing agent. Contrary, the increase in the HF caused decrease in the release of the Ni$^{2+}$ ions from the RhNiTi wires.

The linear regression models for prediction of the release of Ni$^{2+}$ ions from the HF concentration were significant for all three wire types (Table 2). Prediction was strong for the NiTi ($r=0.948$) and the NNiTi ($r=0.940$), but moderate for the RhNiTi ($r=−0.605$; $p<0.05$). Increase of the HF concentration for 1 ppm causes increase of the Ni$^{2+}$ ion release for 0.582 µg/cm$^2$ for the NiTi, and 0.622 µg/cm$^2$ for the NNiTi wire (Table 2). Contrary, the increase of the HF concentration for...
Table 1  Correlation between nickel (Ni\(^{2+}\)) and titanium (Ti\(^{4+}\)) ion release from the uncoated (NiTi), rhodium coated (RhNiTi) and nitrified (NNiTi) wires and the pH, fluoride (F\(^{-}\)) concentration and hydrofluoric acid (HF) concentration determined with the Pearsons correlations

| Wire type | Ni\(^{2+}\) (µg/cm²) | Ti\(^{4+}\) (µg/cm²) | pH | F\(^{-}\) (ppm) | HF (ppm) |
|-----------|----------------------|----------------------|----|----------------|---------|
| NiTi      | 0.873*               | —                    | −0.302 | 0.752*     | 0.948*  |
|           | Ti\(^{4+}\) (µg/cm²) | —                    | −0.302 | 0.506       | 0.797*  |
|           | pH                   | —                    | —    | −0.108      | −0.325  |
|           | F\(^{-}\) (ppm)      | —                    | —    | —           | 0.787*  |
| RhNiTi    | 0.394                | —                    | 0.130 | −0.555      | −0.605* |
|           | Ti\(^{4+}\) (µg/cm²) | —                    | 0.481 | −0.207      | −0.187  |
|           | pH                   | —                    | —    | −0.108      | −0.325  |
|           | F\(^{-}\) (ppm)      | —                    | —    | —           | 0.787*  |
| NNiTi     | 0.850*               | —                    | −0.324 | 0.652*     | 0.940*  |
|           | Ti\(^{4+}\) (µg/cm²) | —                    | −0.070 | 0.396       | 0.788*  |
|           | pH                   | —                    | —    | −0.108      | −0.325  |
|           | F\(^{-}\) (ppm)      | —                    | —    | —           | 0.787*  |

*p<0.05

Table 2  Linear regression models for prediction of nickel ion release from the uncoated (NiTi), rhodium coated (RhNiTi) and nitrified (NNiTi) wires in correlation with the hydrofluoric acid (HF) concentration

| Model    | Unstandardized coefficient B | Std. Error | Standardized coefficient Beta | p   |
|----------|------------------------------|------------|------------------------------|-----|
| NiTi     | Constant                     | 0.970      | 2.757                        | —   |
|          | HF (ppm)                     | 0.582      | 0.062                        | 0.948 |<0.001 |
| RhNiTi   | Constant                     | 36.266     | 10.170                       | —   |
|          | HF (ppm)                     | −0.547     | 0.228                        | −0.605 |0.037  |
| NNiTi    | Constant                     | 0.110      | 3.180                        | —   |
|          | HF (ppm)                     | 0.622      | 0.071                        | 0.940 |<0.001 |

NiTi: R=0.948; R\(^2\)=0.899; Adj. R\(^2\)=0.889
RhNiTi: R=0.605; R\(^2\)=0.366; Adj. R\(^2\)=0.303
NNiTi: R=0.940; R\(^2\)=0.884; Adj. R\(^2\)=0.873

Table 3  Linear regression models for prediction of titanium ion release from the uncoated (NiTi), rhodium coated (RhNiTi) and nitrified (NNiTi) wires in correlation with the hydrofluoric acid (HF) concentration

| Model    | Unstandardized coefficient B | Std. Error | Standardized coefficient Beta | p   |
|----------|------------------------------|------------|------------------------------|-----|
| NiTi     | Constant                     | 0.992      | 0.505                        | —   |
|          | HF (ppm)                     | 0.047      | 0.011                        | 0.797 |0.002  |
| RhNiTi   | Constant                     | 1.568      | 0.433                        | —   |
|          | HF (ppm)                     | −0.006     | 0.010                        | −0.187 |0.561  |
| NNiTi    | Constant                     | 0.570      | 0.533                        | —   |
|          | HF (ppm)                     | 0.048      | 0.012                        | 0.788 |0.002  |

NiTi: R=0.797; R\(^2\)=0.636; Adj. R\(^2\)=0.599
RhNiTi: R=0.187; R\(^2\)=0.035; Adj. R\(^2\)=0.062
NNiTi: R=0.788; R\(^2\)=0.622; Adj. R\(^2\)=0.584
Table 4  Average daily nickel (Ni\textsuperscript{2+}) ion release from the three types of orthodontic wires immersed in different media through periods of time (Ni\textsuperscript{2+} µg/day)

| Wire type | Media type  | Ni\textsuperscript{2+} µg/day |
|-----------|-------------|-------------------------------|
|           |             | 1st–3rd day | 4th–7th day | 8th–14th day | 15th–21st day | 22nd–28th day |
| NiTi      | AS+MiPaste  | 1.13        | 0           | 0.11         | 0.21          | 0.30          |
|           | AS+Elmex    | 12.32       | 2.82        | 5.70         | 13.05         | 0             |
|           | AS+Mirafluor| 5.69        | 2.74        | 4.52         | 3.86          | 25.00         |
| RhNiTi    | AS+MiPaste  | 27.04       | 0.46        | 0.90         | 13.17         | 0.11          |
|           | AS+Elmex    | 0.22        | 0.25        | 0.25         | 0.11          | 0.98          |
|           | AS+Mirafluor| 0.48        | 0.33        | 0.20         | 0.25          | 0.13          |
| NNiTi     | AS+MiPaste  | 0.73        | 0.28        | 0.13         | 0.04          | 0.22          |
|           | AS+Elmex    | 5.44        | 3.56        | 3.41         | 2.83          | 14.94         |
|           | AS+Mirafluor| 6.24        | 6.23        | 8.28         | 6.44          | 21.58         |

NiTi, uncoated nickel-titanium wire; RhNiTi, rhodium coated nickel-titanium wire; NNiTi, nitrified nickel-titanium wire; AS, artificial saliva; Mi Paste, MI Paste Plus; Elmex, Elmex gelée; Mirafluor, Mirafluor-k-gel.

Average daily Ni\textsuperscript{2+} ions release
Average daily Ni\textsuperscript{2+} ions release during the observed period was calculated for two wires (for upper and lower dental arch), for all three wire types and three prophylactic agents (Table 4).

RhNiTi wire had highest average daily Ni\textsuperscript{2+} ions release during the first three days after immersion to the prophylactic agent with low HF concentration, followed by the third week, after immersion to the same agent. After immersion to the prophylactic agents with high HF concentration there was very low average daily Ni\textsuperscript{2+} ions release through all time periods.

NiTi wire had highest average daily Ni\textsuperscript{2+} ions release during the fourth week of use of Mirafluor-k-gel, followed by the fourth week and the first three days of usage of Elmex gelée. The Mi Paste Plus caused highest average daily Ni\textsuperscript{2+} ions release during the first three days, and still it was lower than through any other period of time for the agents with high HF concentration.

NNiTi wire exhibited average daily Ni\textsuperscript{2+} ions release in trends similar to those of NiTi wires (Table 4).

Surface characterization
Micromorphology of the wires' surface showed the most pronounced surface irregularities for specimens immersed to the media that caused low ion release (Figs. 2 a, d, e), when compared to the media which caused high ion release (Figs. 2 b, c, f).
Table 5  Chemical composition of three types of orthodontic wires immersed in different media determined with the energy-dispersive spectroscopy analysis

| Wire type | Media          | Chemical composition, wt% |
|-----------|----------------|-------------------------|
|           |                | Nickel | Titanium | Rhodium | Gold | Carbon | Oxygen |
| NiTi      | AS             | 52.84  | 44.23    | —       | —    | 2.71   | 0.22   |
| NiTi      | AS+Mirafluor   | 51.85  | 43.53    | —       | —    | 4.28   | 0.34   |
| NiTi      | AS+MIPaste     | 52.98  | 43.04    | —       | —    | 3.98   | —      |
| NiTi      | AS+Elmex       | 54.38  | 43.97    | —       | —    | 1.65   | —      |
|             |                |        |          |          |      |        |        |
| RhNiTi    | AS             | 12.21  | 5.63     | 28.40    | 46.19| 5.47   | 2.1    |
| RhNiTi    | AS+Mirafluor   | 5.53   | 0        | 35.76    | 55.21| —      | 3.5    |
| RhNiTi    | AS+MIPaste     | 10.31  | 6.22     | 32.99    | 43.3 | 5.58   | 1.6    |
| RhNiTi    | AS+Elmex       | 9.01   | 5.57     | 29.08    | 50.5 | 4.47   | 1.37   |
| NNiTi     | AS             | 51.74  | 44.51    | —       | —    | 3.75   | —      |
| NNiTi     | AS+Mirafluor   | 49.96  | 42.68    | —       | —    | 7.36   | —      |
| NNiTi     | AS+MIPaste     | 52.04  | 44.74    | —       | —    | 3.22   | —      |
| NNiTi     | AS+Elmex       | 49.71  | 41.81    | —       | —    | 7.29   | 1.19   |

NiTi, uncoated nickel-titanium wire; RhNiTi, rhodium coated nickel-titanium wire; NNiTi, nitrified nickel-titanium wire; AS, artificial saliva; Mi Paste, MI Paste Plus; Elmex, Elmex gelée; Mirafluor, Mirafluor-k-gel.

Table 5 showed distribution of surface and subsurface elements, with more of the nickel being present, when compared to titanium. Also, the coating on RhNiTi wire showed notable presence of gold, but also nickel and titanium.

**DISCUSSION**

Concentration of the HF is the best predictor of the influence of various remineralizing agents on the release of Ni\(^{2+}\) and Ti\(^{4+}\) ions from the uncoated NiTi, as well as rhodium coated and nitride coated NiTi wires. The pH and the F\(^-\) of the prophylactic remineralizing agents influence the corrosion of the various dental materials\(^{24,31,32}\), but the HF concentration should be used to give more accurate prediction for the release of ions from the NiTi alloys.

Findings from the EDS indicate that the surface coating on RhNiTi is not homogenous, because the presence of both surface and subsurface elements was noted. The degree of corrosion of the basic material under protective coating is determined by the quantity and size of the surface defects, as well as the coating thickness\(^{33}\). The release of the Ni\(^{2+}\) and Ti\(^{4+}\) ions should be attributed to the pores found in disrupted surface coating\(^2,3\), and the variability in ion release should be attributed to greater variations in surface coating thickness and homogeneity between the specimens. The TiN coating on NiTi wire is resistant to corrosion in the low HF concentration agent, but repeated administration of the remineralizing agents with high HF causes deterioration of the wire's corrosion resistance, even if the contact between the wire and the remineralizing agent lasts only five min a week. The TiO\(^2\) coating on the uncoated NiTi wire behaves in the similar fashion. In the high HF concentration groups, the highest release of ions was during the fourth week of the experiment for both NiTi and NNiTi wire. This indicates the progressive degradation of the protective surface layer, caused by the repeated application of the remineralizing agents. Contrary, in artificial saliva alone the release of ions from uncoated NiTi decreases with the prolonged time of immersion, because the protective oxide layer remains stable\(^3,18,19\).

The daily dosage of 5 µg of nickel which induce epithelial proliferation\(^{34}\), was observed for the NiTi wire during the first three days, and during the third week when using prophylactic agents with high HF concentration. For the NNiTi wire, epithelial proliferation could be expected during the first three days, and during the first three weeks when using prophylactic agents with high HF concentration. More than 10 µg of nickel a day have cytotoxic effect\(^{35}\), and such cytotoxic daily dosage was observed for the NiTi wire during the first three days and later during the fourth week when using prophylactic agents with high HF concentration. For the NNiTi wire, cytotoxic daily dosage of nickel could be achieved during the fourth week of application of the prophylactic agents with high HF concentration. Cytotoxic effect of nickel could also be expressed during the first three days and through the third week when the RhNiTi wire is in weekly contact with the prophylactic agent with low HF concentration.

Previous researches confirm the importance of the nickel concentration and time of exposure on influence on the cell growth\(^{35,36}\).

Nickel can be found in the environment (water, food, jewellery, kitchen appliances, money coins, wire...
ACKNOWLEDGMENTS

Many thanks to Associate Professor Helena Otmacic Curkovic for her help in conducting this study.

This work was supported by the Croatian Science Foundation grant number IP-2014-09-7500.

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