Original Article

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Effect of fluoride-containing products on nickel-free orthodontic brackets

Natsinee Pipatvadekul, **Kanlaya Insee**, Samroeng Inglam

Faculty of Dentistry, Thammasat University, Pathumthani, Thailand

Objectives: To investigate the nickel ion release, surface morphology, and surface roughness of nickel-free orthodontic brackets after exposed to different fluoride-containing products.

Materials and methods: Twelve samples of nickel-free orthodontic brackets were divided into 3 groups and exposed to different fluoride-containing products; fluoride varnish, acidulated phosphate fluoride (APF) solution, or artificial saliva without fluoride as a control group. The exposure time was following the recommended time of each product. The brackets were exposed to these products following their instructions. Then, all samples were removed from solutions and immersed in artificial saliva for 7 days. The amount of nickel ion and surface roughness were determined with inductively coupled plasma mass spectrometry and a non-contact optical 3-dimensional surface characterization and roughness measuring device, respectively. Their surfaces were evaluated using a scanning electron microscope. The data were analyzed by one-way ANOVA and Dunnett's test at a significance level of 0.05.

Results: The amount of nickel ion released was low and there was no significant difference between control, fluoride varnish, and APF groups (p=0.12). The mean ± SD of nickel ion concentration were irregularities and grooves were presented on the surfaces of nickel-free brackets in fluoride varnish and APF groups. Average surface roughness was higher in the fluoride varnish and APF groups compared to the control group but there were also no significant differences between the three groups (p=0.06).

Conclusion: Fluoride varnish and APF gel have a minor effect on nickel-free orthodontic brackets in the aspect of nickel ion released and surface roughness. These products can be used in orthodontic patients who were treated with fixed nickel-free appliances.

Keywords: bracket, corrosion, fluoride, nickel, surface roughness

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Introduction

Orthodontic appliances are usually made from alloys. In most of these alloys, nickel was incorporated to increase the resistance to corrosion and reduce oxidation at high temperatures [1]. However, nickel is considered to be one of the most common allergens with a contact allergy rate of up to 30% [2, 3], depending on age, gender, and ethnicity [4]. These alloys are subjected to corrosion when they come into contact with the oral environment and release metal ions into the oral cavity, including nickel ions.

Generally, highly biocompatible orthodontic materials are considered for bracket manufacturing. However, some literature has reported side effects, including allergic and inflammatory reactions, cytotoxicity, and mutagenicity [5, 6]. Nickel-induced hypersensitivity involved an inflammation process that promoted the activation and recruitment of immune cells in the area of exposure. Moreover, a study has shown that nickel is a cause of changes in allergic patients' periodontal and immunological status [7].

In the orthodontic bracket manufacturing process, alternative low-nickel or nickel-free alloys have been used to substitute the traditional stainless

Correspondence author: Kanlaya Insee
Faculty of Dentistry, Thammasat University
99 Mu 18 Khlong Luang, Pathumthani 12120, Thailand
Tel.: +66 2986 9213 Email: ikanlaya@tu.ac.th
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steel alloy, especially for people with nickel hypersensitivity. Nickel-free brackets that are defined as stainless steel with low nickel content (less than 5 percent) are claimed to release low nickel ion levels, which may decrease hypersensitivity among allergic patients [8]. Costa MT and the co-workers [9] compared corrosion and cytotoxicity between stainless steel and low-nickel stainless steel brackets in artificial saliva. The result showed that low-nickel stainless steel brackets demonstrated higher corrosion resistance. Ortiz AJ and the co-workers [10] immersed brackets from different alloys in the minimum essential medium. They found that lower amounts of ions were released from nickel-free alloys to medium. However, there were few studies about the corrosion resistance of nickel-free orthodontic brackets.

The corrosion process of metal brackets is linked to their mechanical properties' deterioration and adverse biological effects that can lead to the increase of friction and plaque accumulation. Orthodontic brackets remained in the oral cavity for a long period of an average of 1 to 3 years. Hence, the corrosion resistance of orthodontic alloys is affected by the oral environment. Many factors influence the oral environment such as quantity and quality of saliva, pH of food and drink, temperature, proteins, bacterial flora, enzyme activity, and fluorides [11].

Good oral hygiene is an important part of successful orthodontic treatment. Many orthodontists prescribe a topical fluoride treatment to address inadequate hygiene. Nevertheless, corrosion resistance of orthodontic alloys or metal brackets could be negatively affected by fluoride prophylactic agents. Many researchers have reported that the presence of fluorides could decrease the corrosion resistance of orthodontic alloys [12-19].

To date, there have been no reports of the effects of prophylactic fluoride agents on the nickel-free orthodontic brackets. Therefore, the purposes of this study are to evaluate the amounts of nickel ion released, surface morphology, and surface roughness evaluation of nickel-free brackets exposed to different fluoride products: fluoride varnish and acidulated phosphate fluoride.

Materials and methods

Sample preparation

Nickel-free orthodontic brackets (Forestadent, Pforzheim, Germany) were used in this study. The alloy composes of ≤ 0.10% carbon, ≤ 1.0% silicon, 16% to 20% manganese, 16% to 20% chromium, 1.8% to 2.5% molybdenum, ≤ 0.3% nickel, ≤ 0.05% phosphorus, and ≤ 0.05% sulfur [10]. The bracket slot size is 0.022 inches. A semi-micro-analytical balance (Denver instrument TB-214) was used to weigh the brackets.

Twelve sets of 20 brackets and 4 molar tubes were prepared and divided into 3 groups including varnish group, acidulated phosphate fluoride (APF) group, and control group (artificial saliva).

Fluoride and control solutions

Three solutions were prepared: varnish, APF, and artificial saliva as a control. The artificial saliva was composed of 0.75 g of potassium chloride, 0.07 g of magnesium chloride, 0.199 g of calcium chloride, 0.965 g of dipotassium hydrogen phosphate, 0.439 g of potassium dihydrogen phosphate, 6 g of sodium carboxymethylcellulose, and 2.4 g of sodium benzoate in 1,200 mL of deionized water [15].

The fluoride agents were fluoride varnish (Colgate® Duraphat®), containing 22,600 ppm fluoride and 1.23% APF gel (60 Second Taste®), containing 12,300 ppm fluoride. The APF solution is the mixture of 1.23% APF gel and artificial saliva at a 1:1.4 (v/v) ratio [15]. The pH of fluoride varnish and APF solution was measured by pH meter (FiveEasy Plus FEP20, Mettler Toledo, Switzerland).

The twelve samples were divided into three groups by solution; varnish, APF gel, and control. The samples in each group were exposed to fluoride-containing products in different duration followed by the recommendation of the manufacturer [15].

1. Varnish groups: 4 samples were single applied with a thin coating of fluoride varnish with the aid of a microbrush applicator and immersed in artificial saliva for 30 minutes.

2. APF groups: 4 samples were immersed in the APF solution (5 mL of APF gel mixed with 6.77 mL of artificial saliva) for 4 minutes and dried for 30 minutes.

3. Control groups: 4 samples were immersed in artificial saliva without fluoride for 30 minutes.

Then, the samples were removed from their respective solutions, cleaned with deionized water, and immersed in 8 mL of artificial saliva, at 37 °C for 7 days.

Nickel ion measurement

The concentration of nickel ion release from the brackets was determined by using the inductively coupled plasma mass spectroscopy (ICP-MS) (NexION 350X, PerkinElmer, USA). The ICP-MS has a detection limit of 0.1 µg/L for a nickel. The mean of the measurements was calculated in µg/L.

After 7 days of immersion, 5 mL of solution from each group were collected and 50 microliters of 65% HNO_3 were added to stabilize the ions. The remaining solution was used to re-measure the pH value by a pH meter.

Scanning electron microscope (SEM) analysis

At the end of the procedure, the samples were cleaned in deionized water in an ultrasonic cleaner at 40 kHz for 30 minutes, wiped and left until dry, and scanned by SEM (JEOL BenchTop SEM JCM-6000) at 15 kV and 500x magnification at the slot floor.

Surface roughness evaluation

A non-contact optical 3-dimensional (3D) surface characterization and roughness measuring device (Infinitefocus SL, Alicona, Graz, Austria) was used to measure bracket slot roughness at 50x magnification. A scanned area was approximately 400 x 400 mm^2 around the bracket slot floor (Figure 1). An IF-MeasureSuite software was used to determine the surface roughness (Alicona, Graz, Austria). Each sample was scanned three times and the mean value was calculated.

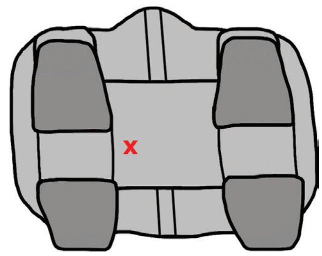

Figure 1 X indicates the area of the brackets scanned by SEM and roughness measuring device

Statistical analysis

The SPSS program version 23.0 was used to analyze the data (SPSS, Chicago, IL). Data were expressed as means and standard deviations. The nickel ion release and surface roughness values passed the normality test of Shapiro–Wilk. The intergroup differences of surface roughness were compared by one-way analysis of variance (ANOVA) and Dunnett Post Hoc analysis. Statistical significance was considered at a *p* level lower than 0.05.

Results

The means and standard deviations of nickel ion release and bracket surface roughness were demonstrated in Table 1.

After immersed in artificial saliva for 7 days, the amount of nickel ion was measured. Concentrations of nickel ions in all groups were quite similar. The highest concentration of nickel ion was detected from the control group, followed by APF and varnish groups, respectively. However, there was no significant difference in nickel ion released between all groups ($p = 0.12$).

Table 1 Concentrations of nickel ion (µg/L) and bracket surface roughness in different solutions (significance level = 0.05)

	Control	Varnish	APF	1-way ANOVA
Nickel ion (µg/L)	4.76 ± 0.31	4.00 ± 0.18	4.06 ± 0.26	p = 0.12
Surface roughness (nm)	461.41 ± 13.79	647.71 ± 70.70	530.79 ± 39.86	p = 0.06

Under SEM at 500x magnification, the surfaces of untreated nickel-free brackets were smoother than those of experimental groups. After immersion for 7 days, brackets in the control group presented slight alteration. For the varnish and APF groups, shallow surface marks and grooves were obviously seen (Figure 2).

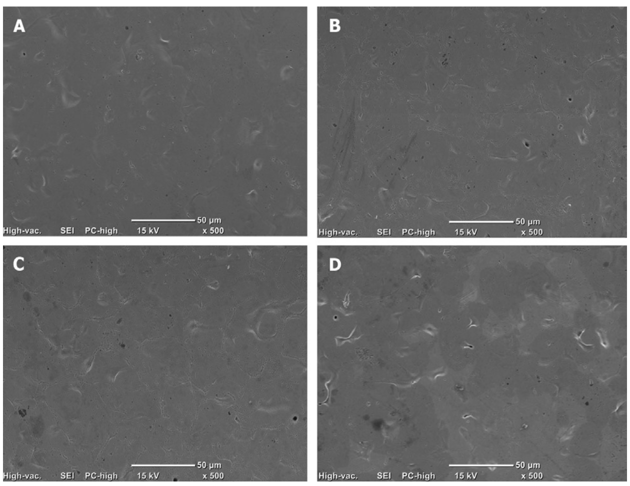

Figure 2 SEM nickel-free bracket surface images (500x magnification); A: untreated bracket, B: control group, C: varnish group, D: APF group

For average surface roughness (Ra), we could notice that surface roughness increased after brackets were exposed to fluoride-containing products. The highest Ra was found in the varnish group, followed by the APF and control groups, respectively. However, there was no significant difference between groups (p = 0.06).

Discussion

The present study evaluated the effect of fluoride-containing products on nickel-free orthodontic brackets in three aspects; nickel ion released, surface morphology, and surface roughness. Each sample contained 20 brackets and 4 molars tubes that represented full-fixed orthodontic appliances used in patients. Fluoride-containing products used in this study were fluoride varnish (22,600 ppm fluoride) and APF gel (12,300 ppm fluoride) that were commonly used in high-risk caries patients to prevent developing white spot lesion or carious lesion. After exposed to fluoride-containing products, the samples were submerged in artificial saliva for 7 days, according to the study of Kuhta M and the co-workers that the highest amount of ions was released during the first week [20].

The corrosion resistance of stainless steel alloys is due to increased chromium concentration. Chromium reacts to oxygen in the environment and then forms a chromium oxide film over the entire steel surface that acts as a block for oxygen spreading into the alloy's body [21]. Thus, oxygen plays an important role in forming and maintenance the oxide film [1], whereas the presence of fluoride ions is detrimental to the protective layer because fluoride ions react with hydrogen ions from bacterial products and acidity of food or drink, then forms hydrofluoric acid which potentially damages the protective oxide layer according to the equation: $Cr_2O_3 + 6HF \rightarrow 2CrF_3 + 3H_2O$ [22]. Therefore, we hypothesized that fluoride-containing product affected nickel-free orthodontic brackets which is one type of the stainless steel alloys.

From the results, nickel ion concentrations from nickel-free brackets were low in all groups and there was no significant difference between groups. Moreover, they were less than the critical value necessary to induce allergy and below the daily dietary intake level (200-300 µg) [23]. Despite methodological differences, the amount of nickel ion released from nickel-free brackets in this study was similar to those from the study of Costa MT and the co-workers that used different brands of nickel-free brackets in their study [9]. Compared to the study of Ortiz AJ and the co-workers, nickel ion concentrations from the samples in this present study were lower than those from their study that immersion for 30 days but did not expose to fluoride-containing products [10]. Yanisarapan T and the co-workers used conventional stainless steel brackets with three types of archwires in their study. They have reported that APF gel can decrease corrosion resistance of fixed metal appliances [15]. Hence, APF gel was included in this present study. After exposed to a fluoride environment, compared to their study, the amount of nickel ions released from nickel-free brackets was dramatically lower than those from conventional stainless steel and there were also no significant differences between groups. These could imply that the corrosion resistance of nickel-free brackets was higher than those of stainless steel brackets, and are in agreement with previous studies [9, 10, 24]. Our findings have shown that fluoride varnish and APF gel had minor effects on nickel-free brackets in the nickel ion released aspect.

From SEM observation, we could observe the smooth surface of untreated nickel-free brackets. The surface morphologies of the brackets exposed to the fluoride varnish and APF showed more irregular patterns. Previous studies [14, 21, 25] have proven that the presence of fluorides in artificial saliva reduced the corrosion resistance of stainless steel. The present study used the non-contact surface profilometer to measure the surface roughness. The advantages of this device are no sample preparation and non-destructive [26]. The quantitative roughness results, corresponding to the qualitative roughness, showed that Ra increased after nickel-free brackets exposed to fluoride-containing products. The highest Ra was presented in the fluoride varnish group. This finding could be caused by the high concentration of fluoride ion in fluoride varnish and some residual fluoride varnish that was sticky and remained on the bracket slot floor. Hence, we suggested that fluoride varnish should be carefully applied to the teeth and minimal directly applied to brackets or archwires.

The sample size in this present study was calculated using G Power analysis software based on the data from Yanisarapan T and the co-workers [15]. Although there was no statistically significant difference in surface roughness between groups, a high standard deviation was presented and the *p*-value almost reached statistical significance ($p = 0.06$). Therefore, increasing the sample size is suggested to narrow the standard deviation and confirm the results.

The results from this study demonstrated that nickel-free brackets presented high corrosion resistance in a fluoride environment in an aspect of nickel ion released and surface roughness. Although fluoride varnish and APF gel could generate increased bracket surface roughness, there were no significant differences. Consequently, nickel-free appliances are good alternative devices for nickel hypersensitive orthodontic patients who need topical fluoride supplements.

Conclusion

A low concentration of nickel ion was detected after nickel-free brackets were exposed to fluoride-containing products and immersed. The quantitative surface roughness was higher in the fluoride varnish and APF group compared to the control group, but not statically different. Thus, fluoride-containing products have a minor effect on nickel-free orthodontic brackets in the aspect of nickel ion released and surface roughness. According to this study, we suggest that fluoride varnish and APF gel can be used in orthodontic patients treated with fixed nickel-free appliances.

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Conflict of Interest: All authors report no conflict of interest related to this study.

References

1. House K, Sernetz F, Dymock D, Sandy JR, Ireland AJ. Corrosion of orthodontic appliances--should we care? *Am J Orthod Dentofacial Orthop* 2008; 133: 584-92.
2. Golz L, Papageorgiou SN, Jager A. Nickel hypersensitivity and orthodontic treatment: a systematic review and meta-analysis. *Contact Dermatitis* 2015; 73: 1-14.
3. Romaguera C, Vilaplana J, Grimalt F. Contact stomatitis from a dental prosthesis. *Contact Dermatitis* 1989; 21: 204.
4. Simonsen AB, Deleuran M, Johansen JD, Sommerlund M. Contact allergy and allergic contact dermatitis in children - a review of current data. *Contact Dermatitis* 2011; 65: 254-65.
5. Golz L, Bayer S, Keilig L, Jager A, Stark H, Bourauel C, et al. Possible implications of Ni(II) on oral IL-1beta-induced inflammatory processes. *Dent Mater* 2014; 30: 1325-35.
6. Kolokitha OE, Chatzistavrou E. A severe reaction to ni-containing orthodontic appliances. *Angle Orthod* 2009; 79: 186-92.
7. Pazzini CA, Pereira LJ, Carlos RG, de Melo GE, Zampini MA, Marques LS. Nickel: periodontal status and blood parameters in allergic orthodontic patients. *Am J Orthod Dentofacial Orthop* 2011; 139: 55-9.
8. Pantuzo MC, Zenobio EG, de Andrade Marigo H, Zenobio MA. Hypersensitivity to conventional and to nickel-free orthodontic brackets. *Braz Oral Res* 2007; 21: 298-302.
9. Costa MT, Lenza MA, Gosch CS, Costa I, Ribeiro-Dias F. In vitro evaluation of corrosion and cytotoxicity of orthodontic brackets. *J Dent Res* 2007; 86: 441-5.
10. Ortiz AJ, Fernandez E, Vicente A, Calvo JL, Ortiz C. Metallic ions released from stainless steel, nickel-free, and titanium orthodontic alloys: toxicity and DNA damage. *Am J Orthod Dentofacial Orthop* 2011; 140: e115-22.
11. Boere G. Influence of fluoride on titanium in an acidic environment measured by polarization resistance technique. *J Appl Biomater* 1995; 6: 283-8.
12. Saporeti MP ME, Sales WF. In vitro corrosion of metallic orthodontic brackets: influence of artificial saliva with and without fluorides. *Dental Press J Orthod* 2012; 17: 24.e1-7.
13. Heravi F, Moayed MH, Mokhber N. Effect of fluoride on nickel-titanium and stainless steel orthodontic archwires: an in-vitro study. *J Dent (Tehran)* 2015; 12: 49-59.
14. Schiff N, Grosgogeat B, Lissac M, Dalard F. Influence of fluoridated mouthwashes on corrosion resistance of orthodontics wires. *Biomaterials* 2004; 25: 4535-42.
15. Yanisarapan T, Thunyakitpisal P, Chantarawaratit P-o. Corrosion of metal orthodontic brackets and archwires caused by fluoride-containing products: Cytotoxicity, metal ion release and surface roughness. *Orthodontic Waves* 2018; 77: 79-89.
16. Walker MP, White RJ, Kula KS. Effect of fluoride prophylactic agents on the mechanical properties of nickel-titanium-based orthodontic wires. *Am J Orthod Dentofacial Orthop* 2005; 127: 662-9.
17. Kao CT, Ding SJ, He H, Chou MY, Huang TH. Cytotoxicity of orthodontic wire corroded in fluoride solution in vitro. *Angle Orthod* 2007; 77: 349-54.
18. Schiff N, Boinet M, Morgon L, Lissac M, Dalard F, Grosgogeat B. Galvanic corrosion between orthodontic wires and brackets in fluoride mouthwashes. *Eur J Orthod* 2006; 28: 298-304.
19. Schiff N, Dalard F, Lissac M, Morgon L, Grosgogeat B. Corrosion resistance of three orthodontic brackets: a comparative study of three fluoride mouthwashes. *Eur J Orthod* 2005; 27: 541-9.
20. Kuhta M, Pavlin D, Slaj M, Varga S, Lapter-Varga M, Slaj M. Type of archwire and level of acidity: effects on the release of metal ions from orthodontic appliances. *Angle Orthod* 2009; 79: 102-10.
21. Huang HH. Corrosion resistance of stressed NiTi and stainless steel orthodontic wires in acid artificial saliva. *J Biomed Mater Res A* 2003; 66: 829-39.
22. Alavi S, Farahi A. Effect of fluoride on friction between bracket and wire. *Dent Res J (Isfahan)* 2011; 8(Suppl 1): S37-42.
23. Europe WROf. Air Quality Guidelines - Second Edition. Chapter 6.10 Nickel. 2000.
24. Alvarez K, Hyun SK, Fujimoto S, Nakajima H. In vitro corrosion resistance of Lotus-type porous Ni-free stainless steels. *J Mater Sci Mater Med* 2008; 19: 3385-97.
25. Gioka C, Bourauel C, Zinelis S, Eliades T, Silikas N, Eliades G. Titanium orthodontic brackets: structure, composition, hardness and ionic release. *Dent Mater* 2004; 20: 693-700.
26. Agarwal CO, Vakil KK, Mahamuni A, Tekale PD, Gayake PV, Vakil JK. Evaluation of surface roughness of the bracket slot floor--a 3D perspective study. *Prog Orthod* 2016; 17: 3.