Analysis of biodegradation of orthodontic brackets using scanning electron microscopy

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Abstract

Objective: The purpose of this study was to analyze, with the aid of scanning electron microscopy (SEM), the chemical and structural changes in metal brackets subjected to an in vitro biodegradation process. **Methods:** The sample was divided into three groups according to brackets commercial brand names, i.e., Group A = Dyna-Lock, 3M/Unitek (AISI 303) and Group B = LG standard edgewise, American Orthodontics (AISI 316L). The specimens were simulated orthodontic appliances, which remained immersed in saline solution (0.05%) for a period of 60 days at 37°C under agitation. The changes resulting from exposure of the brackets to the saline solution were investigated by microscopic observation (SEM) and chemical composition analysis (EDX), performed before and after the immersion period (T0 and T5, respectively). **Results:** The results showed, at T5, the formation of products of corrosion on the surface of the brackets, especially in Group A. In addition, there were changes in the composition of the bracket alloy in both groups, whereas in group A there was a reduction in iron and chromium ions, and in Group B a reduction in chromium ions. **Conclusions:** The brackets in Group A were less resistant to in vitro biodegradation, which might be associated with the type of steel used by the manufacturer (AISI 303).

Keywords: Corrosion. Biocompatibility. Orthodontic brackets. Nickel.

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INTRODUCTION

Over the past 20 years, the biocompatibility of dental alloys has been the target of extensive research. However, studies in this area have generated many unanswered questions, confirming the need to learn much more about the biocompatibility of these materials. Given the fact that this process is not thoroughly understood, orthodontists are hard pressed to select a biologically safe alloy for their patients.

Hypersensitivity caused by nickel in stainless steel alloys, widely employed in orthodontic treatment,[4,20] has become increasingly frequent. Orthodontic brackets, bands and archwires are universally made from this alloy, which contains about 6% to 12% of nickel and 15% to 22% of chromium.[24] Besides allergenicity, carcinogenic, mutagenic and cytotoxic effects have been attributed to nickel and, to a lesser extent, chromium.

One of the factors that determine the biocompatibility of alloys used in dentistry is their resistance to corrosion.[19,27] However, despite the high resistance of austenitic stainless steel, the major alloy employed in the manufacture of orthodontic brackets, several studies have revealed the corrosion of these brackets.[3,9,13,16,18,28,29] The very bracket manufacturing process exposes them to physical and chemical factors that stimulate corrosion. Noteworthy, among these, are thermal treatment,[12] welds[5] and polishing agents.[17]

Macroscopically, bracket corrosion is characterized by loss of gloss, discoloration and superficial roughness often associated with the deposition of products of corrosion.[30] These features, when present, can contribute to increased frictional resistance and interfere with orthodontic mechanics, affecting treatment progress.[11]

According to Edie, Andreasen and Zaytoun,[7] the observation of surface characteristics in order to detect corrosion constitutes the most straightforward method to evaluate biodegradation. It is worth noting that the methodology used in this study to evaluate the homogeneity of the metal matrix, i.e., visual analysis of microscopic images, has proved effective for such evaluation. Chappard et al[6] found a positive relationship between levels of roughness measured by contact profilometry and roughness analysis in microscope images (SEM).

In view of the wide array of factors associated with corrosion and the susceptibility of orthodontic brackets to this process, the purpose of this study was to analyze, using scanning electron microscopy (SEM), the chemical and structural changes in two brands of metal brackets subjected to a process of biodegradation in vitro.

MATERIAL AND METHODS

Microscopic bracket analysis (SEM)

Two different brackets were analyzed: Dyna-Lock Standard Edgewise (3M Unitek, Monrovia, CA, USA) and LG Edgewise (American Orthodontics, Sheboygan, Wisconsin, USA), which were divided into two experimental groups, according to their commercial brands names (Table 1). For evaluation by SEM (Philips XL30,

GROUP	BRACKETS					
	n	Brand	Specification	Type of steel	Chemical composition (max%)	Remark
A	140	3M/ Unitek	Dynalock, Standard Edgewise, Slot 0.022-in	AISI 303	C=0.15%, Chr=17-19%, Ni=5.0-10%, Mn=2.0%, Si=1.0%, Iron=remainder	No welding joining body to base
B	140	American Orthodontics	LG Standard Edgewise, Slot 0.022-in	AISI 316L	C=0.030%, Chr=16-18%, Ni=10-14%, Mn=2.0%, Si=1.0%, Iron=remainder	Silver solder joining body to base

TABLE 1 - Division of the experimental groups.

Eindhoven, Netherlands) 70 brackets were randomly selected and analyzed in two stages: T0 (analyzed "as received") and T5 (60 days after immersion in saline solution).

The specimens that simulated a hemi-mandible consisted of incisor (n = 2), canine (n = 1) and pre-molar (n = 2) brackets. Upper incisor brackets were used on the molars (1st and 2nd), totaling 7 brackets. The brackets were attached to archwires with elastic ligature and the bracket bases covered with wax #7. This procedure was meant to prevent corrosion in that region and facilitate the removal of bonding material from the bracket bases after experiment completion. The specimens were immersed in test tubes containing 10 ml of saline solution (NaCl 0.05%, Biochemistry Department, PUCRS) and subjected to a process of "chemical-mechanical aging". They remained under agitation for 8 hours a day at a constant temperature of 36±1°C (Dubnoff Bath, Nova Técnica™) for a period of up to 60 days.

Photomicrographs were taken of the same regions and the same brackets under the same magnification at both times (T0 and T5).

To perform a SEM analysis, the brackets were mounted on stubs and observed by an examiner. The following images were recorded (Fig 1):

1 - Frontal (general) view - whole bracket (50x magnification).

1s - Frontal (specific) view - 2 pre-determined regions of each bracket were observed: Region a, on the left occlusal/incisal wing, and region b, on the left slot (500x magnification).

2 - Inferior (general) view - whole bracket (50x magnification).

2s - Inferior (specific) view - 2 regions were observed on each bracket at 500x (regions a and b) and 2000x (region 2m) magnification.

At T0 the differences in surface finish of the orthodontic brackets in Groups A and B were qualitatively evaluated. In the following step, the images obtained initially (T0) were compared with those obtained after the brackets had remained immersed in saline solution for 60 days (T5).

All images were qualitatively evaluated by a single examiner.

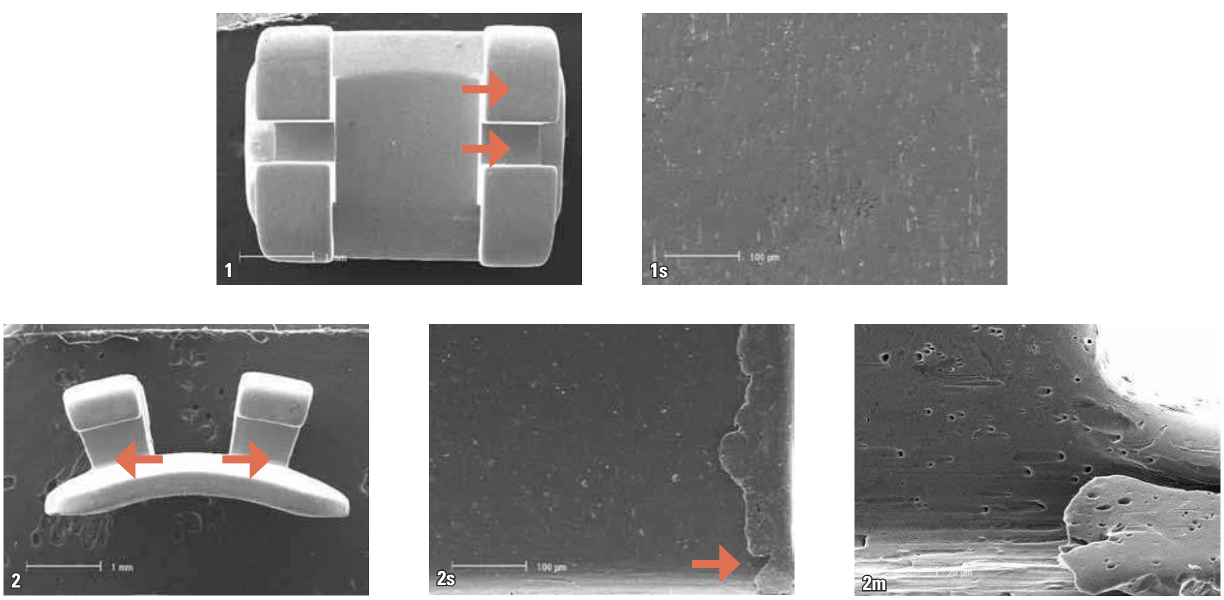

FIGURE 1 - **1**) Frontal image (general): The arrows indicate regions a and b where specific images at 500x magnification were taken. **1s**) Frontal (specific) image. **2**) Inferior image (general): The arrows indicate regions a and b, where specific images at 500x magnification were taken; **2s**, **2m**) Frontal (specific) images at 500x and 2000x magnification, respectively.

Analysis of the chemical composition of the brackets

An EDX (Energy Dispersive X-Ray) was used, which is a SEM resource that allows for the evaluation of the chemical composition of the brackets. SEM procedures were standardized. EDX was performed on 8 brackets for each group, on the buccal and gingival wing surfaces (frontal and inferior images, respectively). It was therefore possible to quantify and compare the iron, nickel and chromium ions found in the metal alloys of the brackets, prior to (T0) and following a 60-day immersion in saline solution (T5).

Statistical treatment

The data gathered from microscopic observation were not treated statistically since such information involved a qualitative comparison between images.

The computer program SPSS version 10.0 (Chicago, IL, USA) was used to analyze the data pertaining to the chemical composition of the brackets. The means for iron, nickel and chromium ions present in the metal alloy of the brackets were compared, "as received" (T0) and after 60 days immersed in saline solution (T5). For intragroup analysis of the EDX values at T0 and T5, the Wilcoxon nonparametric test was used.

RESULTS

Microscopic bracket analysis (SEM)

The microscopic (SEM) analysis at T0 indicated that the brackets in Group A had a better surface finish than those of Group B. Alterations were found on the surfaces of the brackets after a 60-day immersion in saline (T5). These changes were more evident in Group A (Fig 2).

In the frontal images, both general and specific (50x and 500x magnification), products of corrosion were identified in both groups. These products appeared in three different manners, i.e., in a pinhead shape, in clusters and in layers. Group A brackets displayed most often a cluster and layer formation, i.e., their surfaces seemed more altered than the surfaces of Group B brackets (Figs 2 and 3). EDX was performed on the products of corrosion and showed that they were primarily composed of iron (48.82%), oxygen (19.56%), chromium (17.9%) and nickel (4.73%).

On the other hand, an analysis of the inferior images, both general and specific, indicated that the regions most significantly affected in Group A were the wing edges, especially the angle formed between the wing and the bracket base. Regarding the brackets in Group B, the weld regions located between the base and the wing were the most affected by the corrosive process (Fig 4).

Analysis of the chemical composition of the brackets

As shown in Figures 5 and 6, differences were found in the composition of the metal alloy used in the brackets before (T0) and after having remained 60 days immersed in saline solution (T5). The brackets in Group A showed a reduction in the amount of iron and chromium ($p < 0.05$) and the brackets in Group B showed a decrease in chromium ions ($p < 0.05$).

DISCUSSION

Microscopic bracket analysis (SEM)

The superficial homogeneity of the metal alloy is an important factor in the prevention of corrosion pits and cracks.[2,21] Rough surfaces with numerous imperfections facilitate the corrosion process and increase the area of metal dissolution.[2,15].

The role of the bracket manufacturing process in corrosion should be emphasized. Group A brackets are manufactured in one piece (monobloc) using one single type of metal alloy. Group B brackets, in turn, are manufactured in 2 pieces (body and base) joined by silver solder. According to Maijer and Smith[23] the solder used in bracket manufacture appears to be a significant factor in the onset of the corrosion process. In 2001, Lee

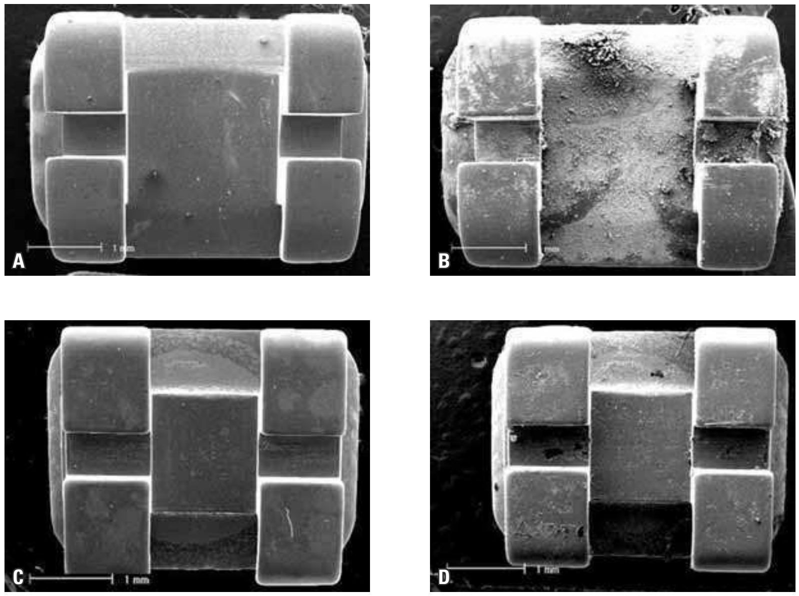

FIGURE 2 - General view (50x) of the brackets in Group A at T0 (**A**) and T5 (**B**) and general view (50x) of the brackets in Group B at T0 (**C**) and T5 (**D**). Products of corrosion can be seen at T5, notably in Group A brackets.

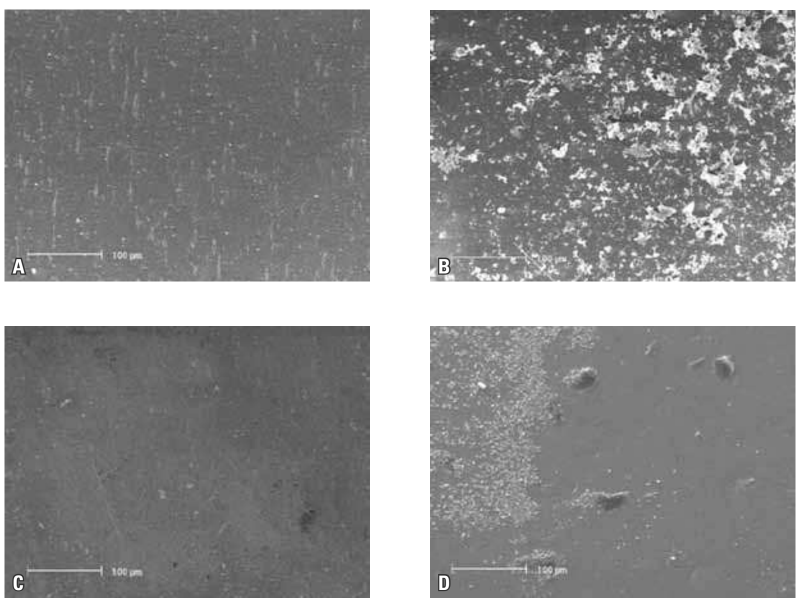

FIGURE 3 - Frontal (specific) images of Group A brackets at T0 and T5 (**A** and **B** respectively) and frontal (specific) images of Group B brackets at T0 and T5 (**C** and **D** respectively). Products of corrosion can be seen at T5, notably in Group A brackets.

and Chang[22] found that heating orthodontic wires (NiTi and Optimalloy) to 250°F for 20 minutes leads them to develop an increased number of pits, worsening corrosion.

Thus, Group B brackets seem to be more susceptible to corrosion because they displayed a greater number of metal matrix irregularities beyond the silver solder used to join bracket body to bracket base. However, after a 60-day immersion, the microscopic images indicated an increased concentration of products of corrosion in the Group A brackets (Figs 2, 3 and 4). It is believed

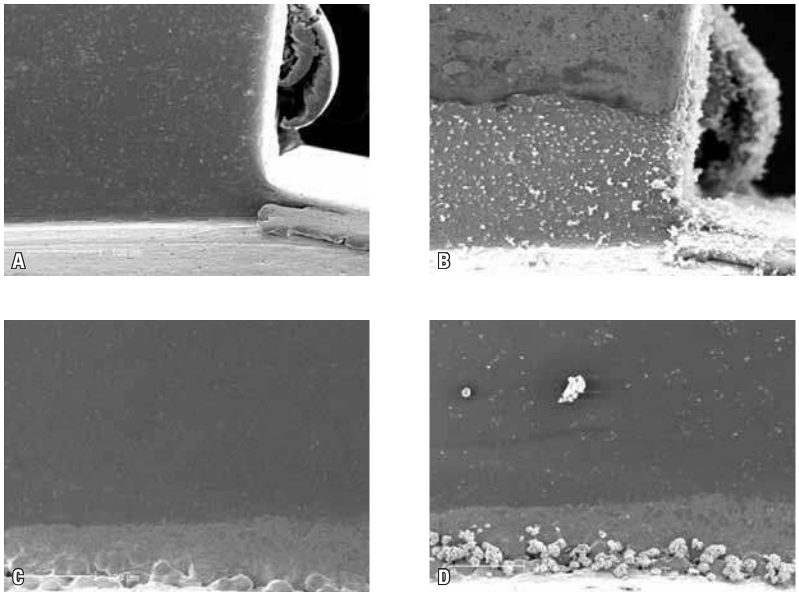

FIGURE 4 - Inferior (specific) images (500x) of Group A brackets at T0 and T5 (**A** and **B** respectively) and inferior (specific) images of Group B brackets at T0 and T5 (**C** and **D** respectively). Products of corrosion can be seen at T5, notably in Group A brackets.

that this result is linked to the composition of alloys used in the different groups: Group A (AISI 303) and Group B (AISI 316L).

It should be emphasized that although the biodegradation of the Group B brackets is less intense, the silver solder area was the most affected by the corrosive process (Fig 4), in agreement with previous studies.[5,12]

Moreover, we observed at T5 that the brackets in Group A often showed the formation of superficial corrosion layers. It is assumed that such corrosion layers is one stage in the dynamics of the corrosive process. In 2000, Oliveira et al[26] emphasized that the corrosive process begins with the penetration of electrolytes into irregularities in the metal matrix (pits and cracks), which react with the metal and form oxides/hydroxides that accumulate gradually. The results of this study seem to confirm this corrosive process dynamics, suggesting the occurrence of a corrosion cycle of metal brackets, which is determined by the following events: 1) Filling of pits by products of corrosion, 2) formation of clusters of products of corrosion on the surface of the brackets, 3) layers of products of corrosion covering specific parts of the bracket surface, 4) removal of corrosion layers from the surface (probably due to mechanical factors) and the start of a new corrosion cycle. In this last stage changes can be observed in the anatomy of the metal brackets.

It is essential to bear in mind that, in this study, the regions most affected by corrosion were those that exhibited some type of defect in the metal matrix, corroborating with other studies.[2,17,21,25] This seems to prove that a pronounced surface roughness is a predisposing factor to the corrosion process since it tends to increase the contact area between the metal matrix and the immersion solution. Furthermore, Grimsdottir and Hensten-Pettersen[15] emphasized that the surface defects noted in nickel-titanium orthodontic wires are not large enough to act as corrosion-prone areas. This seems to be a controversial issue and, therefore, it should be reminded that the corrosive process is determined by multiple factors.[1,14,16]

Analysis of the chemical composition of the brackets

The EDX is a SEM tool that allows us to identify and quantify the metals comprised in an alloy, and this identification is roughly proportional to the fractions by weight of each element. Thus, we can measure the release of nickel, chromium and iron in an indirect fashion. According to Eliades et al[10] this method has clinical relevance and achieves results with a significant degree of reliability.

An analysis of alloy composition indicated that the brackets in Group A, analyzed "as received", had amounts of iron, nickel and chromium ions compatible with those described for the composition of AISI 303 steel. On the other hand, Group B brackets (AISI 316L), analyzed "as received", showed an amount of nickel ions lower than that quantity established for this type of steel. This lower content of nickel in the alloy could affect characteristics such as ductility, weldability and corrosion resistance.

At T5, we found a significant reduction of iron and chromium ions in Group A alloy and decreased chromium ions in Group B alloy (Figs 5 and 6). These data are consistent with the findings obtained by microscopic analysis, whereby Group A brackets were also the most extensively affected.

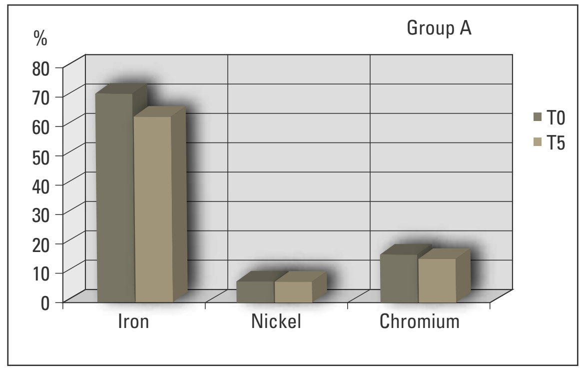

FIGURE 5 - Chemical composition (EDX) of Group A bracket alloy at T0 and T5. There was a reduction in the amount of iron ($p < 0.05$) and chromium ($p < 0.05$) ions.

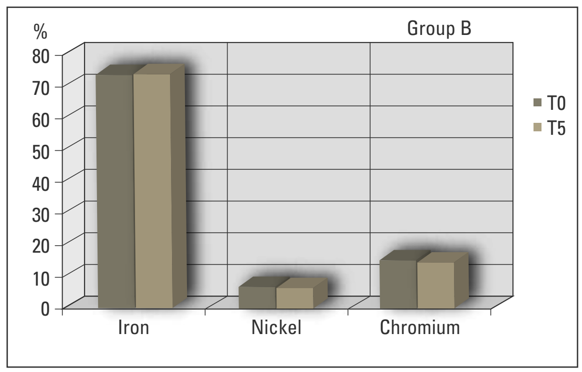

FIGURE 6 - Chemical composition (EDX) of the Group B bracket alloy at T0 and T5. There was a reduction in the amount of chromium ions ($p < 0.05$).

FINAL CONSIDERATIONS

Despite numerous studies investigating the ionic release of orthodontic brackets, no conclusive evidence has yet been produced with respect to the kinetics and composition of corrosive products.[8] It should be noted that the use of alloys with lower biodegradability would reduce the risk of harm to patient health. Therefore, researchers have been trying to investigate the main factors that determine the corrosive process.

The alloy and manufacturing process of orthodontic brackets seem to play an important role in their corrosion resistance.[13] The fact remains that the relationship between corrosion and biocompatibility of orthodontic appliances seems to be an issue that is still far from settled in the literature. Therefore, the findings of this study concerning the biodegradation of orthodontic brackets should not be discarded as negligible or clinically insignificant, since further investigations are needed to explain this phenomenon.

CONCLUSIONS

Based on the results of this study we concluded that:

a) Using SEM, we observed the presence of products of corrosion on the brackets, especially in Group A. The regions most affected were those that showed some irregularity of the metal matrix.

b) An analysis of the chemical composition of the brackets, prior to (T0) and following the in vitro experiment (T5), revealed changes in the ratio of ions. In Group A, a decrease in iron and chromium ions, and in Group B, a reduction of chromium ions, after immersion (T5).

REFERENCES

1. Arvidson K, Johansson EG. Galvanic series of some dental alloys. Scand J Dent Res. 1977 Sep;85(6):485-91.
2. Azevedo CRF. Characterization of metallic piercings. Eng Failure Anal. 2003 Jun;10(3):255-63.
3. Barrett RD, Bishara SE, Quinn JK. Biodegradation of orthodontic appliances. Part I. Biodegradation of nickel and chromium in vitro. Am J Orthod Dentofacial Orthop. 1993 Jan;103(1):8-14.
4. Bass JK, Fine H, Cisneros GJ. Nickel hypersensitivity in orthodontic patient. Am J Orthod Dentofacial Orthop. 1993 Mar;103(3):280-5.
5. Berge M, Gjerdet NR, Erichsen ES. Corrosion of silver soldered orthodontic wires. Acta Odontol Scand. 1982;40(2):75-9.
6. Chappard D, Degasne I, Huré G, Legrand E, Audran M, Baslé MF. Image analysis of roughness by texture and fractal analysis correlate with contact profilometry. Biomaterials. 2003 Apr;24(8):1399-407.
7. Edie JW, Andreasen GF, Zaytoun MP. Surface corrosion of nitinol and stainless steel under clinical condition. Angle Orthod. 1981 Oct;51(4):319-24.
8. Eliades T, Athanasiou AE. In vivo aging of orthodontic alloys: implications for corrosion potential, nickel release, and biocompatibility. Angle Orthod. 2002 Jun;72(3):222-37.
9. Eliades T, Eliades G, Watts DC. Intraoral aging of the inner headgear component: a potential biocompatibility concern? Am J Orthod Dentofacial Orthop. 2001 Mar;119(3):300-6.
10. Eliades T, Trapalis C, Eliades G, Katsavrias E. Salivary metal levels of orthodontic patients: a novel methodological and analytical approach. Eur J Orthod. 2003 Feb;25(1):103-6.
11. von Fraunhofer JA. Corrosion of orthodontic devices. Semin Orthod. 1997 Sep;3(3):198-205.
12. Gjerdet NR, Hero H. Metal release from heat-treated orthodontic archwires. Acta Odontol Scand. 1987 Dec;45(6):409-14.
13. Grimsdottir MR, Gjerdet NR, Hensten-Pettersen A. Composition and in vitro corrosion of orthodontic appliances. Am J Orthod Dentofacial Orthop. 1992 Jun;101(6):525-32.
14. Grimsdottir MR, Hensten-Pettersen A. Citotoxic and antibacterial effects of orthodontic appliances. Scand J Dent Res. 1993 Aug;101(4):229-31.
15. Grimsdottir MR, Hensten-Pettersen A. Surface analysis of nickel-titanium arch wire used in vivo. Dent Mater. 1997 May;13:163-7.
16. Huang TH, Yen CC, Kao CT. Comparison of ion release from new and recycled orthodontic brackets. Am J Orthod Dentofacial Orthop. 2001 Jul;120(1):68-75.
17. Hunt NP, Cunningham SC, Golden CG, Sheriff M. An investigation into the effects of polishing on surface hardness and corrosion of orthodontic arch wires. Angle Orthod. 1999 Oct;69(5):433-40.

18. Hwang CJ, Shin JS, Cha JY. Metal release from simulated fixed orthodontic appliances. Am J Orthod Dentofacial Orthop. 2001 Oct;120(4):383-91.
19. Jones TK, Hansen CA, Singer MT, Kessler HP. Dental implications of nickel hypersensitivity. J Prosthet Dent. 1986 Oct;56(4):507-9.
20. Kerosuo H, Kullaa A, Kerosuo E, Kanerva L, Hensten-Pettersen A. Nickel allergy in adolescents in relation to orthodontic treatment and piercing of ears. Am J Orthod Dentofacial Orthop. 1996 Feb;109(2):148-54.
21. Kim H, Johnson JW. Corrosion of stainless steel, nickel-titanium, coated nickel-titanium, and titanium orthodontic wires. Angle Orthod. 1999 Feb;69(1):39-44.
22. Lee SH, Chang YI. Effects of recycling on the mechanical properties and the surface topography of nickel-titanium alloy wires. Am J Orthod Dentofacial Orthop. 2001 Dec;120(6):654-63.
23. Maijer R, Smith DC. Biodegradation of the orthodontic bracket system. Am J Orthod Dentofacial Orthop. 1986 Sep;90(3):195-8.
24. Matasa CG. Attachment corrosion and is testing. J Clin Orthod. 1995 Jan;29(1):16-23.
25. Matasa CG. Metallography and you. II. Surface analysis. The Orthodontic Materials Insider. 1998 Dec;11(4):1-7.
26. Oliveira JC, Cavaleiro A, Brett CMA. Influence of sputtering conditions on corrosion of sputtered W-Ti-N thin film hard coatings: salt spray tests and image analysis. Corrosion Science. 2000 Mar;42:1881-95.
27. Schmalz G, Garhammer P. Biological interactions of dental cast alloys with oral tissues. Dent Mater. 2002 Jul;18(5):396-406.
28. Sória ML. Avaliação da corrosão de bráquetes metálicos. [dissertação]. Rio Grande do Sul: Universidade Federal de Pelotas; 2003.
29. Sória ML, Menezez L, Dedavid B, Pires M, Rizzatto S, Costa Filho LC. Avaliação in vitro da liberação de níquel por bráquetes metálicos. Rev Dental Press Ortod Ortop Facial. 2005 maio-jun;10(3):87-96.
30. Toms AP. The corrosion of orthodontic wire. Eur J Orthod. 1998 May;10(2):87-97.

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