An *in vitro* comparison of nickel and chromium release from brackets

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Abstract: This study aimed at comparing amounts of nickel (Ni) and chromium (Cr) released from brackets from different manufacturers in simulated oral environments. 280 brackets were equally divided into 7 groups according to manufacturer. 6 groups of brackets were stainless steel, and 1 group of brackets was made of a cobalt-chromium alloy with low Ni content (0.5%). International standard ISO 10271/2001 was applied to provide test methods. Each bracket was immersed in 0.5 ml of synthetic saliva (SS) or artificial plaque fluid (PF) over a period of 28 days at 37°C. Solutions were replaced every 7 days, and were analyzed by spectrometry. The Kruskal-Wallis test was applied. Amounts of Ni release in SS ($\mu g\ L^{-1}$ per week) varied between groups from "bellow detection limits" to 694, and from 49 to 5,948.5 in PF. The group of brackets made of cobalt-chromium alloy, with the least nickel content, did not release the least amounts of Ni. Amounts of Cr detected in SS and in PF ($\mu g\ L^{-1}$ per week) were from 1 to 10.4 and from 50.5 to 8,225, respectively. It was therefore concluded that brackets from different manufacturers present different corrosion behavior. Further studies are necessary to determine clinical implications of the findings.

Descriptors: Orthodontic brackets; Nickel; Chromium; Saliva, artificial.

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Introduction

The corrosion process of metallic brackets has been linked to the deterioration of their mechanical properties and to adverse biological effects.[1-8] Since none of these aspects are desirable in orthodontic practice, comparing amounts of metal release from commercially available brackets is necessary to determine their resistance to corrosion in the oral environment.

The American Iron and Steel Institute (AISI) types 316L or 304 austenitic stainless steel alloys are currently used for bracket manufacturing.[9,10] These steel alloys typically contain approximately 8% nickel (Ni) and 18% chromium (Cr) with a small amount of manganese and silicon, and a low carbon content (less than 0.1%).[9,10] AISI type 316L also contains 2 to 3% molybdenum.[9,10] Besides that, bracket manufacturing includes different processes with or without welding. Therefore, some brackets may be a layered complex of alloys differing in composition and mechanical state as various parts may be welded or brazed together.[10]

As a group, the cobalt-base alloys may be generally divided in three categories described as wear-resistant, corrosion-resistant and heat-resistant materials.[11] Cobalt-base wear-resistant alloys contain the least Ni content (3% max), 25 to 30% Cr, 0.25 to 3.3% carbon, and also manganese, silicon, molybdenum, tungsten, iron and sodium.[11] This alloy is used in bracket manufacturing. However, although the cobalt-base wear-resistant alloys (with low Ni content) exhibit some resistance to aqueous corrosion, it is limited.[11] To satisfy the industrial need for alloys that exhibit higher resistance to aqueous corrosion, it was necessary to increase Ni content (9 to 35%), and decrease carbon content (0.8% max) in the cobalt-base corrosion-resistant alloys.[11] The third category of cobalt-base alloys, the high-temperature alloys, is used in industry.[11]

This study aimed at comparing amounts of Ni and Cr released from various bracket models from different manufacturers in simulated oral environments over a period of 28 days.

Material and Methods

The sample comprised 280 orthodontic brackets of upper premolars from the MBT™ prescription.[12] The brackets were equally divided into 7 groups from different models and manufacturers as follows: Kirium Line™ Abzil™ (São José do Rio Preto, SP, Brazil – code: 288-133), Mini Master Series™ American Orthodontics™ (Sheboygan, WI, USA – code: 390-0027), Discovery™ Dentaurum™ (Ispringen, Baden-Württemberg, Germany – code: 790-118-00), Full Size™ Unitek™ (Monrovia, CA, USA – code: 119-936), Morelli M.B.T.™ Morelli™ (Sorocaba, SP, Brazil – code: 10-35-007), NuEdge™ TP Orthodontics™ (LaPorte, IN, USA – code: 293-205), and Victory™ Unitek™ (Monrovia, CA, USA – code: 017-890). The different brackets studied were labeled A to G respectively according to the model/manufacturer. The brackets were made of stainless steel (approximately 8% Ni and 18% Cr), except for the F brackets which were of cobalt-chromium alloy with low Ni content (0.5% Ni).[9,11] The brackets were tested in an "as-received" state, and complied with the requirement of "no visible signs of change or deterioration". The base of the brackets was not covered with resin, thus eliminating the possibility of extraneous sources of Ni and Cr.

Brackets from each manufacturer were divided into four groups of 10 specimens. An initial corrosion test was carried out on 10 brackets from each manufacturer immersed in synthetic saliva, and 10 brackets from each manufacturer immersed in artificial plaque fluid. An identical corrosion test was performed on the other 20 brackets immersed in the same solutions 30 days later.

International standard ISO 10271/2001, "Dental metallic materials – corrosion test methods", was applied to provide test methods.[13]

Ni and Cr release from brackets, comprising the sample, was quantified by means of a static immersion test. The studied brackets had no contact with metallic materials during the test and each bracket was placed in a separate polypropylene tube (Axigen™, Union City, CA, USA) containing 0.5 ml of synthetic saliva or artificial plaque fluid. The simulated saliva medium was synthesized on the basis of the formula of Leung and Darvell.[14] The final pH was 6.7 ± 1. Artificial plaque fluid was prepared by dissolving 10.0 ± 0.1 g 90% (m m^{-1}) $C_3H_6O_3$ and

5.85 ± 0.005 g NaCl in approximately 300 ml of water, and then by adjusting volume to 1,000 ± 10 ml with distilled water. The final pH was 2.3 ± 1. The container was closed to prevent evaporation of the solution, and the sample tubes were stored at 37° C for 28 days. Every 7 days ± 1 h brackets were removed from each tube, and placed in other tubes with fresh immersion solution. Furthermore, 3 tubes containing the solution prepared at each experimental period, but with no brackets, were used as controls, and were stored exactly as the sample tubes were.

The solutions inside each tube at each experimental period were analyzed by spectrometry to determine Ni and Cr content. All synthetic saliva samples and artificial plaque fluid samples from controls and from C and G brackets were analyzed by simultaneous graphite furnace atomic absorption spectrometry (SIMAAS), model SIMAA 6000 (Perkin Elmer Life and Analytical Sciences™, Shelton, CT, USA), equipped with longitudinal Zeeman-effect background correction, Echelle optical arrangement, and solid-state detector. All solutions were fed into the graphite tube by means of an AS-72 autosampler (Perkin Elmer Life and Analytical Sciences™). Argon 99.996% (v v^{-1}) (White Martins™, São Paulo, SP, Brazil) was used as the purge gas. The instrumental setting-up conditions are shown in Table 1. Artificial plaque fluid samples from A, B, D, E, and F brackets presented high concentrations of Ni and Cr. In this case, determination of Ni and Cr was not possible by SIMAAS, since several dilutions would have been necessary, decreasing accuracy. Therefore, levels of Ni and Cr were determined by inductively coupled plasma optical emission spectrometry (ICP OES), model Spectro Ciros CCD™ (Spectro Analytical Instruments GmbH & Co.™, Kleve, Germany). The instrumental setting-up conditions are shown in Table 2. The detection limits of SIMAAS and ICP OES were calculated based on calibration curves. For SIMAAS, the detection limits for the synthetic saliva samples (µg L^{-1}) were 2.78 for Ni and 0.27 for Cr; for the artificial plaque fluid samples (µg L^{-1}), the detection limits were 2.77 for Ni and 0.85 for Cr. For ICP OES, the detection limits were the same (0.4 µg L^{-1}) for both elements.

The Ni and Cr released values from 7 different groups of brackets immersed in 2 different solutions over a period of 28 days were analyzed using the Kruskal-Wallis test and the non-parametric multiple comparison test. Tests were performed with a 5%

Table 1 - Instrumental setting for determination of Cr and Ni release in synthetic saliva and artificial plaque fluid by SIMAAS-6000.

Element			Cr	Ni
Wavelength			232.0 nm	357.0 nm
Band pass			0.7 nm	0.7 nm
Lamp type*			HCL	HCL
Lamp current			25 mA	25 mA
Calibration range			2.5 to 20 µg L^{-1}	0.1 to 0.8 µg L^{-1}
Signal measurements			Peak area (AA-BG)	
Standard and sample volume			10 µl	
Standard and sample replicates			3	
Heating program for SIMAAS				
Step	T (°C)	Ramp (s)	Hold (s)	Ar (mL min^{-1})
Dry I	110	10	15	250
Dry II	130	1	10	250
Pyrolysis	1,300	10	20	250
Atomization**	2,400	0	5	0
Cleaning	2,500	1	3	250

*HCL: Hollow Cathode Lamp; **Read time.

level of significance. Values below the detection limits were subject to statistical analysis, even if inaccurate, having been estimated by the apparatus. In the tables, however, these values are referred to as "below detection limits" (<DL).

Table 2 - ICP operating conditions.

Generator	Free-running at 27.12 MHz
Power (W)	1400
Nebulizer	Cross-flow (Spectro)
Spray chamber	Double pass, Scott-type
Outer gas (L min^{-1})	12
Intermediate gas (L min^{-1})	1.0
Nebulizer gas (L min^{-1})	1.0
Sample uptake rate (ml min^{-1})	1.5
Analytical wavelenght (nm)	Cr (I) 341.476
	Ni (I) 205.552

(I): Atomic emission line.

Results

The mean weekly values for Ni and Cr release (µg L^{-1}) from the studied brackets immersed in synthetic saliva and artificial plaque fluid over the experimental time and the data from the control group are presented in Table 3. Since the Kruskal-Wallis test was applied, median values were considered instead of mean values. The amount of Ni released in synthetic saliva (µg L^{-1} per week) varied between groups from <DL (below detection limits) (C brackets) to 694 (A brackets); in artificial plaque fluid, it varied from 49 (C brackets) to 5,948.5 (E brackets). The group of brackets made of cobalt-chromium alloy (F), with the least nickel content (0.5%), did not release the least amounts of Ni. Amounts of Cr detected in synthetic saliva and in artificial plaque fluid (µg L^{-1} per week) were from 1 (D brackets) to 10.4 (F brackets) and from 50.5 (G brackets) to 8,225 (D brackets), respectively.

The P values obtained from the non-parametric multiple comparison test for Ni and Cr release in

Table 3 - Mean weekly values of Ni and Cr release (µg L^{-1}) for different groups of brackets immersed in synthetic saliva and artificial plaque fluid.

Brackets	Solution	Ni release				Cr release			
		Mean	SD	Median	N	Mean	SD*	Median	N
A	S	597.76	289.49	694.00	80	13.39	11.55	10.00	80
	PF	6,118.95	3,942.51	5,033.50	80	6,733.90	2,134.24	6,395.50	80
B	S	10.39	11.11	6.55	80	5.73	4.57	4.20	80
	PF	1,731.08	982.91	1,566.00	80	3,590.30	2,516.70	3,286.00	80
C	S	6.93	9.42	<DL	80	1.60	1.42	1.05	80
	PF	237.83	446.83	49.00	80	102.60	79.58	74.00	80
D	S	13.07	15.96	6.60	80	2.27	3.09	1.00	80
	PF	6,999.11	6,334.58	4,035.00	80	13,727.13	11,691.77	8,225.00	80
E	S	54.28	46.62	35.85	80	6.84	8.26	3.90	80
	PF	5,960.30	4,390.25	5,948.50	80	6,867.76	4,607.71	7,381.50	80
F	S	14.17	15.08	8.95	80	13.17	7.43	10.40	80
	PF	1,570.48	1,221.76	1,514.50	80	2,472.35	1,958.00	2,314.50	80
G	S	25.26	25.83	17.10	80	4.36	5.12	1.75	80
	PF	197.64	374.94	76.00	80	114.55	207.22	50.50	80
Control	S	<DL	-	<DL	24	0.23	0.33	0.05	24
	PF	<DL	-	<DL	24	0.68	0.72	0.40	24

*SD: Standard deviation; S: Synthetic Saliva; PF: Artificial Plaque Fluid; <DL: Bellow detection limits (2.78 µg L^{-1} for Ni and 0.27 µg L^{-1} for Cr in synthetic saliva and 2.77 µg L^{-1} for Ni and 0.85 µg L^{-1} for Cr in artificial plaque fluid).

synthetic saliva and artificial plaque fluid among the different studied brackets and controls are displayed in Table 4.

Graphs 1 to 4 present median values of Ni and Cr release (μg L^{-1}) in synthetic saliva and artificial plaque fluid detected at each week over the experimental time. These graphs do not show a trend toward decrease or increase in metal release from week 1 to week 4 in general.

Artificial plaque fluid samples from the experimental groups presented significantly higher rates of Ni and Cr than synthetic saliva samples ($p < 0.001^*$). The control group did not present difference between artificial plaque fluid and synthetic saliva samples (p = 0.950 for Ni and 0.585 for Cr).

Discussion

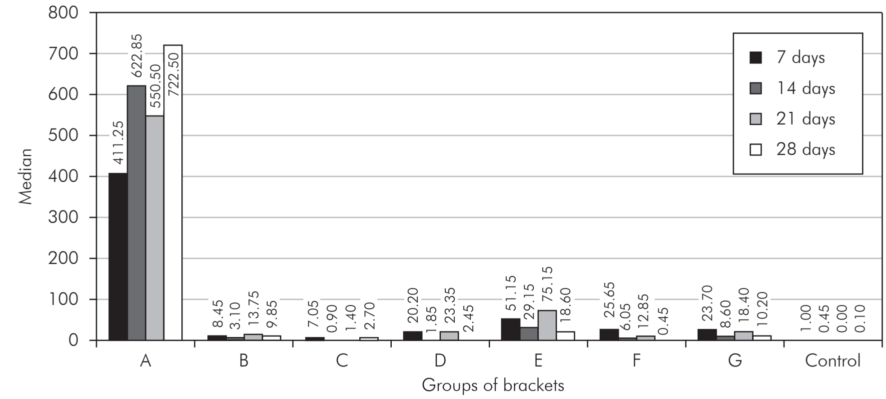
This study has compared the Ni and Cr release from 7 groups of different commercially available brackets in simulated oral environments. The amounts of Ni and Cr released from brackets were quite different among the groups, and varied ac-

Table 4 - P values obtained from nonparametric multiple comparisons of controls and studied brackets for Ni and Cr release (μg L^{-1}) in synthetic saliva and artificial plaque fluid.

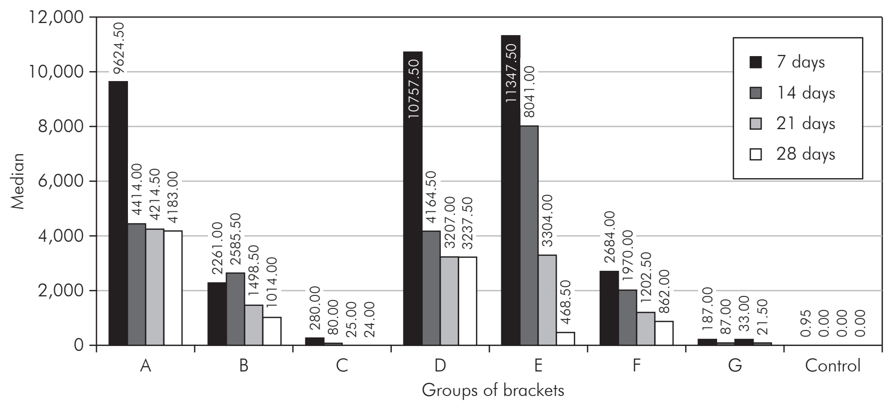
Comparison	Ni release		Cr release	
	Saliva	Plaque Fluid	Saliva	Plaque Fluid
A & Control	< 0.001*	< 0.001*	< 0.001*	< 0.001*
B & A	< 0.001*	0.001*	0.037*	0.014*
B & C	0.148	< 0.001*	0.001*	0.001*
B & Control	0.010*	< 0.001*	< 0.001*	< 0.001*
C & A	< 0.001*	< 0.001*	< 0.001*	< 0.001*
C & Control	0.109	< 0.001*	0.106	< 0.001*
D & A	< 0.001*	0.833	< 0.001*	0.367
D & B	0.950	0.002*	0.002*	0.001*
D & C	0.166	< 0.001*	0.765	< 0.001*
D & Control	0.011*	< 0.001*	0.069	< 0.001*
D & F	0.665	< 0.001*	< 0.001*	< 0.001*
E & A	< 0.001*	0.224	0.025*	0.522
E & B	< 0.001*	0.036	0.878	0.068
E & C	< 0.001*	< 0.001*	0.001*	< 0.001*
E & Control	< 0.001*	< 0.001*	< 0.001*	< 0.001*
E & D	< 0.001*	0.315	0.003*	0.123
E & F	< 0.001*	0.003*	0.005*	0.029*
E & G	0.025*	< 0.001*	0.187	< 0.001*
F & A	< 0.001*	< 0.001*	0.584	0.005*
F & B	0.711	0.354	0.009*	0.722
F & C	0.069	< 0.001*	< 0.001*	0.004*
F & Control	0.005*	< 0.001*	< 0.001*	< 0.001*
G & A	< 0.001*	< 0.001*	< 0.001*	< 0.001*
G & B	0.047*	< 0.001*	0.140	< 0.001*
G & C	0.001*	0.874	0.048*	0.671
G & Control	< 0.001*	< 0.001*	0.003*	< 0.001*
G & D	0.040*	< 0.001*	0.094	< 0.001*
G & F	0.105	< 0.001*	< 0.001*	0.001*

*Statistically significant difference at P < 0.05.

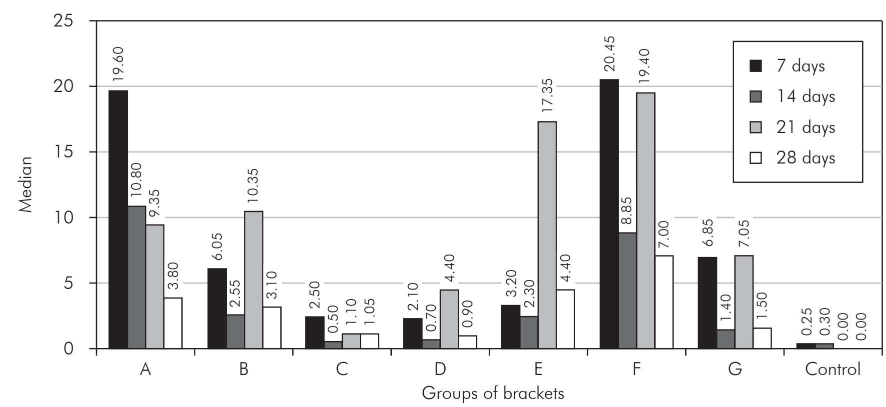
Graph 1 - Median values of Ni concentration (μg L⁻¹) detected in synthetic saliva for controls and studied brackets.



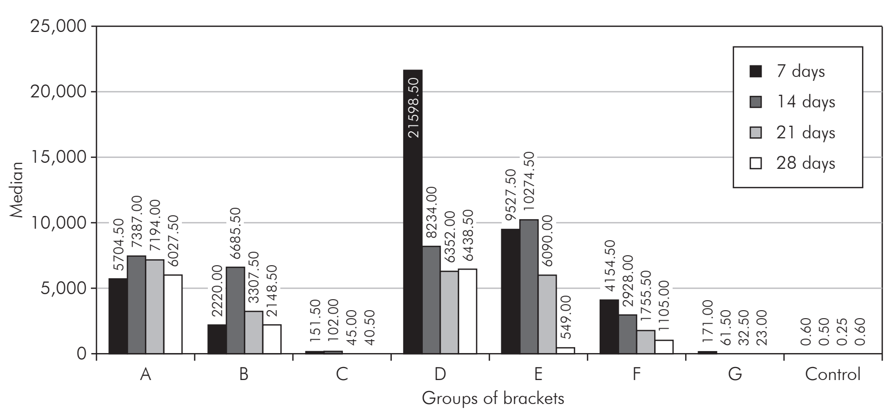
Graph 2 - Median values of Ni concentration (μg L⁻¹) detected in artificial plaque fluid for controls and studied brackets.



Graph 3 - Median values of Cr concentration (μg L⁻¹) detected in synthetic saliva for controls and studied brackets.



Graph 4 - Median values of Cr concentration (μg L⁻¹) detected in artificial plaque fluid for controls and studied brackets.



cording to the studied metal (Ni or Cr) or the immersion medium.

Results exhibited high standard deviation and variance values, which seems to be a sample characteristic, also found by Barrett *et al.*[15] (1993) and Eliades *et al.*[16] (2004). However, it was possible to find statistical differences between the groups since the amounts of Ni and Cr release were quite different. The sample size was determined in compliance with ISO 10271/2001.[13] Additionally, this study used a sample size even larger than the sample size used by other *in vitro* studies that measured metal release.[15-18]

A direct comparison between the values obtained in this study and those obtained in other studies cannot be made since different methodologies were applied or different variables were tested. Barrett *et al.*[15] (1993), Hwang *et al.*[19] (2001) and Shin *et al.*[20] (2003) have tested complete orthodontic appliances immersed in different synthetic saliva formulas. Staffolani *et al.*[18] (1999) tested orthodontic appliances immersed in organic and inorganic acids. Eliades *et al.*[16] (2004), Huang *et al.*[21] (2001) and Huang *et al.*[22] (2004) observed smaller values of metal release than this study. However, these studies all used different immersion solutions, and did not include solutions replaced weekly, which may have caused a saturation of the immersion medium, decreasing Ni and Cr release. Platt *et al.*[9] (1997) tested different alloys, fragments of 2205 and 316L stainless steel, but not orthodontic brackets. Kuhta *et al.*[23] (2009) tested metal ion release from simulated orthodontic appliances with different types of archwires, and observed that the type of archwire can also influence the release of ions.

The A brackets presented the highest amounts of Ni release in artificial saliva, and the C brackets presented the least. The F brackets, made of Co-Cr wear-resistant alloy with the least Ni content (0.5%),[11] did not release the least amounts of Ni. This corrosion behavior of the F brackets can be explained by the characteristics exhibited by the Co-Cr wear-resistant alloy with low nickel content (3% max.) in aqueous medium. According to the Key to Metals Database,[11] even though this alloy possesses some resistance to aqueous corrosion, it is limited by grain boundary carbide precipitation, as well as by the lack of vital alloying elements in the matrix, after formation of the carbides, and by chemical segregation in the microstructure. These characteristics are important and clinically relevant.

Comparing Victory™ and Full Size™ from Unitek™, it was evident that different models from the same manufacturer may exhibit different rates of Ni and Cr release.

Graphs 1 to 4 did not present a trend toward increasing or decreasing rates of Ni and Cr release over the experimental period. Defining a pattern of Ni and Cr release over time was not an objective of this study as it would be necessary to conduct a long term study for that. Wataha, Lockwood[24] (1998), which evaluated metal release from alloys in cell culture over 10 months, detected metal release during the whole experiment. Barrett *et al.*[15] (1993) observed a decrease in Ni release over 28 days, and a variation in Cr release during the study. Grimsdottir *et al.*[17] (1992) performed immersion tests for 14 days, and Sfondrini *et al.*[25] (2009) performed them for 120 hours, although ISO 10271/2001[13] determines a minimum observation period of 28 days.

Our results showed that the artificial plaque fluid caused a significantly higher rate of Ni and Cr release than artificial saliva. This finding agrees with the findings of other studies.[18,21-23] It also reinforces the necessity of appropriate oral hygiene to minimize corrosion rates.

Further *in vivo* studies are necessary to determine the clinical implications of the findings of this study.

Conclusion

Based on the results of the present investigation, which have compared the amounts of Ni and Cr released from commercially available brackets, it was concluded that corrosive behavior was different among the various bracket models from different manufacturers. Increasing rates of Ni and Cr release from the studied brackets were observed in the following order: C, G, B, F, D, E and A.

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