

Evaluation of ionic degradation and slot corrosion of metallic brackets by the action of different dentifrices

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Objective: To evaluate the *in vitro* ionic degradation and slot base corrosion of metallic brackets subjected to brushing with dentifrices, through analysis of chemical composition by Energy Dispersive Spectroscopy (EDS) and qualitative analysis by Scanning Electron Microscopy (SEM).

Methods: Thirty eight brackets were selected and randomly divided into four experimental groups (n = 7). Two groups (n = 5) worked as positive and negative controls. Simulated orthodontic braces were assembled using 0.019 x 0.025-in stainless steel wires and elastomeric rings. The groups were divided according to surface treatment: G1 (Máxima Proteção Anticáries[®]); G2 (Total 12[®]); G3 (Sensitive[®]); G4 (Branqueador[®]); Positive control (artificial saliva) and Negative control (no treatment). Twenty eight brushing cycles were performed and evaluations were made before (T₀) and after (T₁) experiment. **Results:** The Wilcoxon test showed no difference in ionic concentrations of titanium (Ti), chromium (Cr), iron (Fe) and nickel (Ni) between groups. G2 presented significant reduction (p < 0.05) in the concentration of aluminium ion (Al). Groups G3 and G4 presented significant increase (p < 0.05) in the concentration of aluminium ion. The SEM analysis showed increased characteristics indicative of corrosion on groups G2, G3 and G4. **Conclusion:** The EDS analysis revealed that control groups and G1 did not suffer alterations on the chemical composition. G2 presented degradation in the amount of Al ion. G3 and G4 suffered increase in the concentration of Al. The immersion in artificial saliva and the dentifrice Máxima Proteção Anticáries[®] did not alter the surface polishing. The dentifrices Total 12[®], Sensitive[®] and Branqueador[®] altered the surface polishing.

Keywords: Orthodontic brackets. Corrosion. Scanning Electron Microscopy.

Objetivo: avaliar *in vitro* a degradação iônica e corrosão do fundo do slot de braquetes metálicos submetidos à escovação com dentifrícios, realizando análises da composição química por Espectroscopia de Energia Dispersiva (EDS) e qualitativa por Microscopia Eletrônica de Varredura (MEV). **Métodos:** foram selecionados 38 braquetes divididos aleatoriamente em quatro grupos experimentais (n = 7). Dois grupos (n = 5) funcionaram como controles positivo e negativo. Aparelhos ortodônticos simulados foram confeccionados com fios de aço inoxidável 0,019" x 0,025" e anéis elastoméricos. Os grupos foram divididos de acordo com o tratamento de superfície: G1 (Máxima Proteção Anticáries[®]); G2 (Total 12[®]); G3 (Sensitive[®]); G4 (Branqueador[®]); Controle Positivo (saliva artificial) e Controle Negativo (sem tratamento). Foram realizados 28 ciclos de escovação e avaliações antes (T₀) e após (T₁) o experimento. **Resultados:** o teste de Wilcoxon indicou não existir diferença nas concentrações iônicas de titânio (Ti), cromo (Cr), ferro (Fe) e níquel (Ni) entre os grupos. O grupo G2 apresentou redução significativa (p < 0,05) na concentração do íon alumínio (Al) e os grupos G3 e G4 apresentaram aumento significativo (p < 0,05) nas concentrações do íon alumínio. A análise em MEV mostrou aumento nas características indicativas de corrosão dos grupos G2, G3 e G4. **Conclusão:** a análise por EDS revelou que os grupos controle e G1 não sofreram alterações na composição química. O grupo G2 apresentou degradação na quantidade de íons Al, e G3 e G4 sofreram aumento na concentração de Al. A imersão em saliva artificial e o dentifrício Máxima Proteção Anticáries[®] não alteraram o polimento de superfície. Os dentifrícios Total 12[®], Sensitive[®] e Branqueador[®] alteraram o polimento de superfície.

Palavras-chave: Braquetes ortodônticos. Corrosão. Microscopia eletrônica de varredura.

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INTRODUCTION

Deterioration and corrosion of orthodontic appliances is a concern for orthodontists in their clinical practice. This attention is focused in two main questions: If the corrosion by products, whether they are produced, are absorbed by the organism and can cause local or systematic effects; and which effects the corrosion has on the physical properties and clinical performance of orthodontic appliances. There are evidences that support the deleterious effects of the buccal environment on structural alterations of metallic biomaterials.²³ Eliades et al^{6,7} tested orthodontic materials and did not find ion release by nickel-titanium alloys, but observed levels of nickel and chromium released by stainless steel alloys. No material was mentioned as cytotoxic, probably due low quantity of released ions or for the way they link.

The several appliances (brackets, arch wires, etc.) used during orthodontic treatment to obtain the dental movement are composed of materials with distinct physical integrity, structural compositions and mechanical properties. Their requirements are complex for they are put under many stresses in the oral environment. This includes immersion in saliva and ingested liquids, temperature variation, mastication efforts and the loading submitted to the appliances. The combination of these materials and hostile conditions provided by the environment can result in corrosion, ion release and deterioration of these appliances. The interactions that these corrosion by products can have on the manifestation of local or systematic problems in the individual's health is not well corroborated and comprehended in literature,^{3,4,11} despite numerous case reports of hypersensitivity to nickel^{13,21} and alterations on the ionic concentration of body fluids.^{1,16} House et al¹¹ through wide literature review concluded, based on the best available evidences, that the corrosion does not seem to be a process that should cause concern. However, he suggests that further studies, in clinically relevant situations, can lead to a better comprehension of corrosion clinical effects.

It is known that a physical characteristic of great interest for orthodontic mechanics is the contact surface rugosity of the wire and the slot, once this can interfere on the superficial friction and, therefore, on the sliding mechanics.^{15,19} Variation on chemical composition and superficial aspect of metallic brackets² and their biodegradation before different stimulus^{5,10} have been described in literature. Studies have shown that, in an

acid environment and in presence of fluoride ions the resistance to corrosion of certain materials may deteriorate.^{24,30} There are findings that show that fluoridated mouthwashes may influence on the resistance to corrosion²⁷ and galvanic corrosion²⁶ of orthodontic brackets as well as on the corrosion and modification of mechanical properties of orthodontic wires.^{12,14,17,29,30}

Few previous studies were performed to evaluate the influence and clinical implications of fluoridated dentifrices on orthodontic brackets, although there are clinical evidences²⁵ and *in vitro* studies^{8,9,18,20,22} of the deleterious effects produced by the challenges that these appliances are submitted to in the oral cavity. Knowing that innumerable dentifrices commonly used by orthodontic patients are commercially available, with the most varied compositions and concentrations of substances that in contact with metal can induce alterations and that these changes on the surface properties of orthodontic brackets may jeopardize the orthodontic mechanics, the objective of this study was to evaluate *in vitro* the ionic degradation and the slot base corrosion of metallic brackets when subjected to mechanical brushing with different dentifrices, through analysis of chemical composition by Energy Dispersive Spectroscopy (EDS) and qualitative analysis through Scanning Electron Microscopy (SEM).

MATERIAL AND METHODS

Selection of accessories and construction of specimens

For the development of this study, 38 orthodontic metallic brackets (austenitic stainless steel linked to Cr-Ni) of premolars, Edgewise prescription, slot 0.22 x 0.28-in, (Morelli Ortodontia[®]), were selected determining a number of specimens compatible to the factors in study originated from pilot study and calculation of sampling error. The accessories were randomly divided into four experimental groups of 7 brackets each. Two groups of 5 brackets worked as positive and negative controls. For each experimental group specimens were made characterizing a simulated orthodontic appliance constituted of 0.019 x 0.025-in stainless steel wires (Morelli Ortodontia[®]) and elastomeric rings (Morelli Ortodontia[®]) to tie the orthodontic brackets to the wire (Fig 1). After making the specimens the groups were divided according to type of treatment to be used on the surface of the orthodontic brackets (Table 1).

The positive control groups G1, G2, G3 and G4 were kept during experimental design in test tubes containing 10 ml of artificial saliva (Fig 2).

Evaluation of the ionic composition of orthodontic metallic brackets before surface treatment. (T_0)

The 38 orthodontic brackets used in study, divided in their respective groups, were subjected to initial quantitative analysis (T_0) aiming to determine the chemical composition by Energy Dispersive Spectroscopy (EDS) in magnification of 70x with 20KV and 500 μ m of distance. Four points on the brackets surface were selected to obtain concentrations of Aluminium (Al), Titanium (Ti), Chromium (Cr), Iron (Fe) and Nickel (Ni), originating a total of 28 points of analysis per experimental group (Fig 3). The percentage of ionic composition were obtained and classified for posterior achievement of the representative composition mean of each group. After classification, the data were submitted to statistical analysis using Bioestat 5.0 software.



Figure 1 - Standardized specimen for the experiment.



Figure 2 - Specimen in test tube containing artificial saliva.

Qualitative analysis through Scanning Electron Microscopy (SEM) of the metallic brackets' slot surface

The topographic analysis of slot base surface characteristics was evaluated in magnification of 500x through Scanning Electron Microscope Leo Zeiss 1450VP (Fig 4). Each one of the 38 microphotographs obtained from the 4 experimental groups and from the 2 control groups were evaluated according to 8 characteristics determined by established criteria² for surface characterization and possible verification of corrosion indications (Table 2).

An evaluator previously calibrated in relation to possible characteristics to be observed on the surface of the slot base, examined the microphotographs in two distinct moments, without knowing their group, aiming to eliminate possible intraevaluator errors. When discrepancy was found between the two evaluations, a third evaluation was performed and this became definitive, rejecting the prior ones. Each characteristic represented 1 score. This way, it can be obtained a value from 0 to 8 for each microphotograph depending on the number of characteristics present on the slot surfaces.

Table 1 - Division according to treatment, chemical composition of the substance used and trade name.

Group	Treatment	Composition	Trade name
Negative control	No treatment	-	-
Positive control	Immersion in artificial saliva	NaH ₂ PO ₄ , NaCl, KCl, CaCl ₂ , Na ₂ S, (NH ₄) ₂ SO ₄ , C ₆ H ₈ O ₇ , NaHCO ₃ , Urea	-
Group 1 (G1)	Mechanic brushing with dentifrice for 15s - 28 days	Fluoride, calcium carbonate, sodium lauryl sulphate, sodium saccharin, tetrasodium pyrophosphate, sodium silicate, polyethylene glycol, sorbitol, carboxymethyl cellulose, Methylparaben, Propylparaben, Flavor and Water, Sodium monofluorophosphate	Colgate Máxima Proteção Anticáries®
Group 2 (G2)	Mechanic brushing with dentifrice for 15s - 28 days	Sodium Fluoride, Triclosan, Water, Glycerin, Sorbitol, hydrated silica, sodium lauryl sulphate, PVM / MA copolymer, aroma, Carrageen, sodium saccharin, sodium hydroxide, Titanium dioxide	Colgate Total 12 Clean Mint®
Group 3 (G3)	Mechanic brushing with dentifrice for 15s - 28 days	Potassium citrate, sodium monofluorophosphate, zinc citrate, sorbitol, water, glycerin, hydrated silica, PEG-12, Tetrapotassium pyrophosphate, PVM / MA copolymer, sodium lauryl sulfate, flavor, potassium hydroxide, Cellulose Gum, Saccharin sodium, xanthan gum, Titanium Dioxide, Polyethylene, Calcium Carbonate, acid Rojo 18, Amarillo # 5 aluminum Lacquer, Rojo 28.	Colgate Sensitive Multi Proteção®
Group 4 (G4)	Mechanic brushing with dentifrice for 15s - 28 days	Calcium carbonate, sorbitol, alumina, sodium lauryl sulphate, aroma, sodium monofluorophosphate, cellulose gum, sodium silicate, sodium bicarbonate, xanthan gum, sodium saccharin, Methylparaben, Propylparaben.	Colgate Ultra Branqueador®

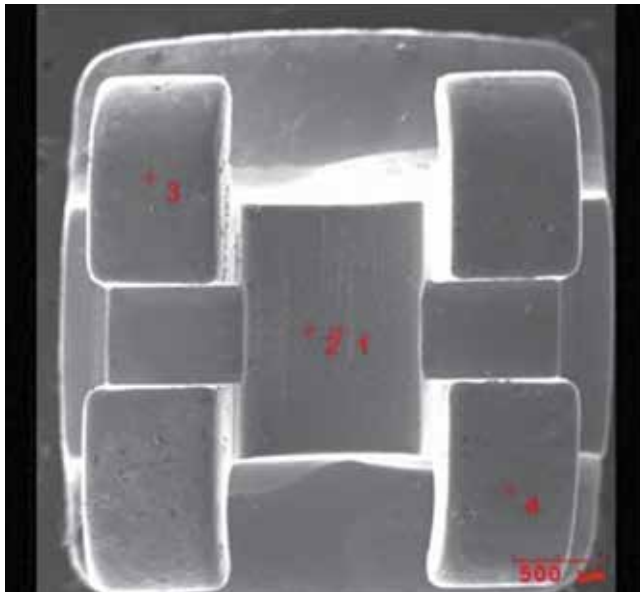


Figure 3 - Points selection and analysis by EDS.



Figure 4 - Leo Zeiss 1450VP Scanning Electron Microscope.

Table 2 - Criteria for qualitative evaluation of metallic bracket slot surface.

Classification	Characteristic
A	Presence of furrow
B	Mottled or spotted aspect
C	Presence of depressions, pores, furrows or grains in less than 1/3 of the surface
D	Presence of depressions, pores, furrows or grains in 1/3 of the surface
E	Presence of depressions, pores, furrows or circumferential grains in 2/3 of the surface
F	Presence of depressions or pores in the entire surface extent
G	Honeycomb aspect
H	Presence of indentations

The obtained data were inserted into forms previously elaborated aiming to classify the results. The most frequent value between the 7 brackets from each group, i.e., mode (measure used for qualitative data) was taken as the group's representative value. The groups that presented the highest value were considered the least polished, while the ones that presented lower value, the most polished.

Experimental design

The specimens correspondent to each group were subjected to surface treatment, which consisted in putting a similar amount of the standardized dentifrice per group on the brush using the transversal technique, mechanic brushing for 15 seconds, always performed by the same operator,²² using the Colgate Motion® electric toothbrush. After mechanic brushing the specimens were rinsed with 20 ml of distilled water during 20 seconds with assistance of a disposable syringe.

Finished the surface treatment the specimens were returned into test tubes containing artificial saliva, being changed at every cycle to avoid bacterial proliferation, and they were kept in room temperature (Fig 5). At every 24 hours it was performed a new cycle of surface treatment. On the positive control groups the appliances were only rinsed in distilled water and the saliva was changed. On the negative control group it was not applied any kind of treatment.

Twenty-eight cycles of surface treatment were performed. By the end of the 28th cycle the specimens were removed from the test tubes containing artificial saliva and rinsed in 20 ml of distilled water for 20 seconds. The orthodontic brackets were carefully removed from the simulated orthodontic braces, removing the elastomeric rings, and submitted to new analysis of chemical composition by Energy Dispersive Spectroscopy (EDS) and qualitative analysis in SEM, after the utilization of dentifrices (T_1).

The descriptive statistical analysis of data was performed to obtain the percentage means and standard deviations of each ionic component on the different groups evaluated. The Wilcoxon test ($\alpha=5\%$) was used to compare variables before (T_0) and after (T_1) the experiment and verify the statistical difference on ionic concentrations.

RESULTS

Table 3 represents the descriptive analysis performed in which the means and standard deviations of ionic concentrations were obtained for the experimental groups and positive control group before (T_0) and after (T_1) experimental design. The evaluation of total ionic concentration showed that there is alteration for groups G2, G3 and G4 after surface treatment (T_1). The comparison through Wilcoxon test showed that there is no statistically significant difference on ionic concentrations of titanium (Ti), chromium (Cr), iron (Fe) and nickel (Ni) between the evaluations T_0 and T_1 . The comparative analysis

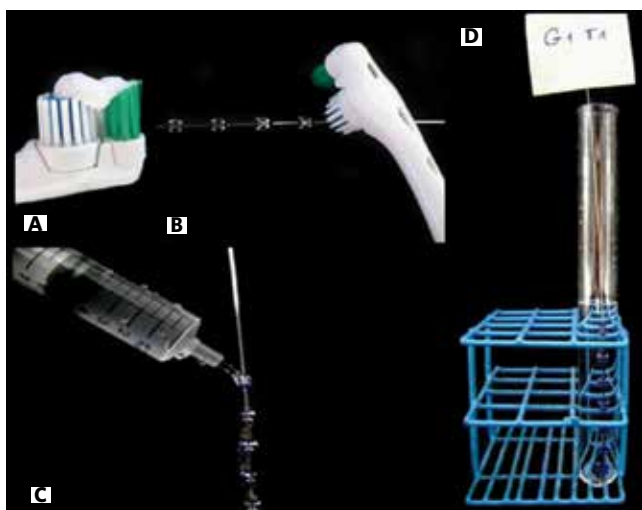


Figure 5 - A) Standardized amount of dentifrice; B) Mechanic brushing; C) Rinsing; D) Storage.

through Wilcoxon test presented statistically significant reduction ($p < 0.05$) for the mean concentration of Aluminium ion (Al) on group G2. Groups G3 and G4 presented statistically significant increase ($p < 0.05$) on the mean concentrations of aluminium considering the comparison between T_0 and T_1 .

Table 4 represents the qualitative analysis of the number of characteristics present on the slot base of each one of the brackets and the mode value (higher frequency) for each experimental group, in T_0 and T_1 . It is observed an alteration on the number of characteristics present, shown by the alteration of modal value, for groups G2, G3 and G4.

The microphotographs representative of each experimental group confirm the surface characteristics determined by the scores (Fig 6). Group G1 according to number of most frequent characteristics, remained equally polished in T_0 and T_1 . Groups G2, G3 and G4 as it can be noticed through representative images and assigned scores (Table 4) presented an increase on the number of surface characteristics, becoming therefore less polished after use of dentifrices.

DISCUSSION

There are evidences of degradation and oxidation of orthodontic metallic appliances in the oral environment.^{1,3,4,8,13,25} The ionic release from the point of view of health's integrity at local and systematic level does not present significant clinical implications according to most publications.^{1,6,11,16} Therefore, studies that point al-

Table 3 - Mean value (%) and standard deviation of the different groups evaluated before and after surface treatment.

	Al	Ti	Cr	Fe	Ni
Group 1 T_0	0.16 ± 0.07	0.12 ± 0.02	18.32 ± 0.13	72.65 ± 0.23	8.76 ± 0.12
Group 1 T_1	0.13 ± 0.13	0.14 ± 0.03	18.46 ± 0.24	72.66 ± 0.23	8.62 ± 0.35
Group 2 T_0	0.24* ± 0.18	0.13 ± 0.02	18.13 ± 0.67	72.96 ± 0.98	8.54 ± 0.42
Group 2 T_1	0.09* ± 0.02	0.13 ± 0.02	18.33 ± 0.18	72.66 ± 0.18	8.79 ± 0.17
Group 3 T_0	0.13** ± 0.05	0.11+ ± 0.02	18.27 ± 0.15	72.76 ± ± 0.12	8.73 ± 0.15
Group 3 T_1	0.20** ± 0.08	0.15+ ± 0.07	18.25 ± 0.09	72.54 ± ± 0.14	8.86 ± 0.08
Group 4 T_0	0.20** ± 0.13	0.15 ± 0.08	18.39 ± 0.21	72.46 ± 0.59	8.81 ± 0.27
Group 4 T_1	4.96** ± 6.40	0.13 ± 0.03	17.57 ± 1.18	68.79 ± 4.69	8.54 ± 0.63
Positive control T_0	0.11 ± 0.05	0.13 ± 0.02	18.31 ± 0.19	72.64 ± 0.31	8.81 ± 0.10
Positive control T_1	0.12 ± 0.04	0.13 ± 0.02	18.28 ± 0.18	72.59 ± 0.19	8.88 ± 0.14
Negative control	0.21 ± 0.19	0.25 ± 0.27	18.26 ± 0.34	72.34 ± 1.02	8.94 ± 0.44

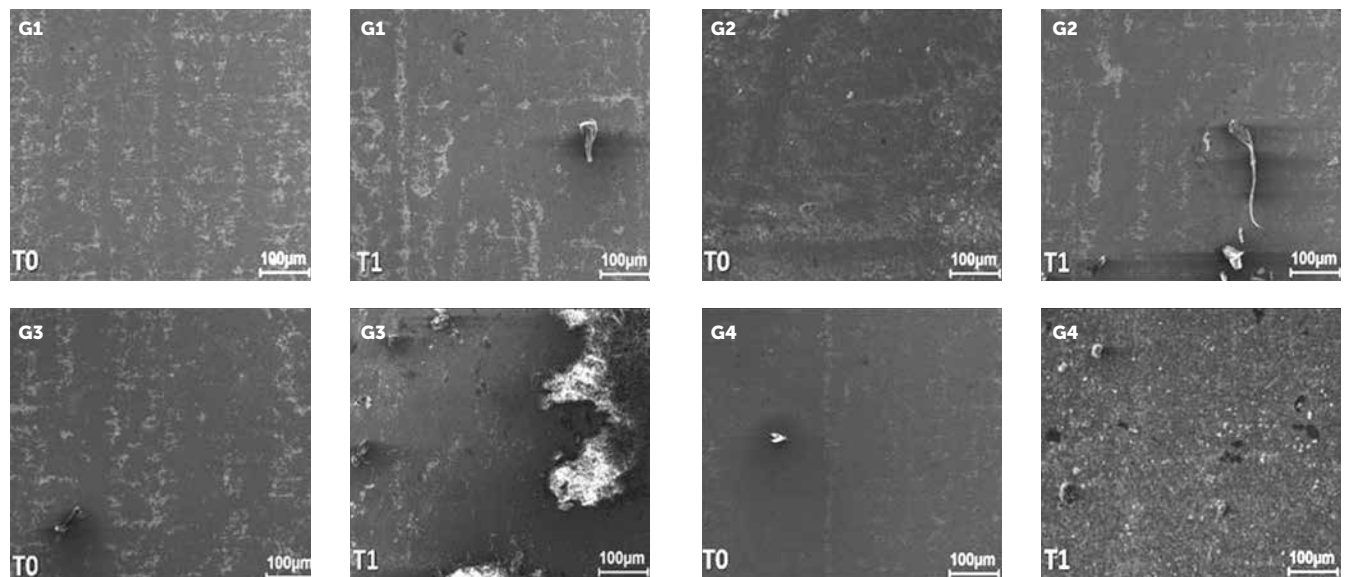
*Indicates significant difference between ions in different times for group 2 ($p < 0.05$).

** Indicates significant difference between ions in different times for group 3 ($p < 0.05$).

*** Indicates significant difference between ions in different times for group 4 ($p < 0.05$).

Table 4 - Number of characteristics present on the brackets' slot base surface for each group and mode (most frequent value) obtained for each group.

	Number of characteristics present on the brackets' slot base														Mode	
	B1		B2		B3		B4		B5		B6		B7		T ₀	T ₁
	T ₀	T ₁	T ₀	T ₁	T ₀	T ₁	T ₀	T ₁	T ₀	T ₁	T ₀	T ₁	T ₀	T ₁	T ₀	T ₁
Negative C	2	-	2	-	3	-	1	-	2	-	-	-	-	-	-	-
Positive C	2	3	3	3	1	2	2	2	2	2	-	-	-	-	-	-
G1	1	2	2	2	1	3	2	2	1	2	2	2	2	2	2	2
G2	2	3	2	2	2	3	3	4	3	3	2	2	3	3	2	3
G3	2	4	2	3	1	2	2	4	1	3	2	3	2	3	2	3
G4	2	3	2	4	1	3	2	4	1	4	2	4	3	4	2	4

**Figure 6** - Microphotographs of brackets' slot base surface representing G1, G2, G3 and G4 in T₀ and T₁ (500x magnification).

lergic reactions, hypersensitivity and possible damages that can be caused by ionic release and corrosion justify the performance of studies that help to better understand the impact of these alterations on the patient's health and on orthodontic treatment.

The results of the present study did not indicate differences on the ionic composition, evaluated by EDS, for the positive control group and G1 (conventional fluoridated dentifrice). G2, which was submitted to brushing with dentifrice containing antimicrobial (Total 12[®]) presented degradation on the mean concentration of Al ions. G3 (Sensitive[®] dentifrice) and G4 (Branqueador[®] dentifrice)

presented an increase on the concentration of these ions, probably due the presence of Aluminium Lacquer on the composition of dentifrice standardized for G3 and Alumina on the composition of dentifrice standardized for G4. These results indicate that elements present on the dentifrices composition can induce unpredictable alterations on the superficial ionic composition of orthodontic metallic brackets. The lack of studies hinders comparison with the obtained results, however, it indicates that new researches must be performed for better identification of structural alterations to which the orthodontic brackets are subjected inside the oral cavity.

These alterations can be of great damage for the orthodontic mechanics, since they can interfere negatively on the friction during sliding mechanics. Groups G2, G3 and G4 presented increase on qualitative characteristics indicative of corrosion, which deserves attention since the lower the surface polishing, more difficult the sliding mechanics,^{15,19} maybe bringing prejudice to the orthodontic treatment.

Schiff et al²⁶ suggests that mouthwashes must be prescribed according to orthodontic materials used, due to the structural alterations that may be induced. This fact agrees with the present work, due to alterations on the superficial composition and on surface characteristics of metallic brackets evaluated both quantitatively and qualitatively.

Based on the results from this study it can be suggested that new studies are performed, in clinically relevant situations, for better comprehension of the factors that can induce alterations on the chemical composition and corrosion of orthodontic appliances and identify how these factors can cause impair to orthodontic mechanics.

CONCLUSION

According to factors evaluated in this research and the methodology applied, the obtained results allow to conclude that:

- a) Analysis of chemical composition by Energy Dispersive Spectroscopy (EDS) revealed that the control group (immersed in artificial saliva) and G1 (Máxima Proteção Anticáries[®]) did not presented alterations on the chemical composition of brackets after experimental design. G2 (Colgate Total 12 Clean Mint[®]) presented degradation on the amount of Al ions, while G3 (Sensitive Multi Proteção[®]) and G4 (Colgate Ultra Branqueador[®]) presented increase on the concentration of Al ions on the brackets surface.
- b) The qualitative analysis through Scanning Electron Microscopy (SEM) revealed that immersion in artificial saliva and brushing using the dentifrice Máxima Proteção Anticáries[®] did not affect the surface polishing of metallic brackets, while the same procedure performed with dentifrices Total 12 Clean Mint[®], Sensitive Multi Proteção[®] and Ultra Branqueador[®] are capable to alter the surface polishing increasing the number of characteristics indicative of corrosion.

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