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The effect of metal surface treatment on bond strength interface between metal/cement

O efeito do tratamento de superfícies metálicas na interface de resistência de união entre metal / cimento

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ABSTRACT

Objective: This study evaluated the hypothesis that different treatments of surface upon three metal alloys for metal ceramic dental prostheses (Gold; Nickel-Chromium; Titanium) do not Influence the values of bond strength with resin cement. **Material and Methods:** Twenty blocks, 5 x 5 x 5 mm, of each alloy were divided into two subgroups (n = 10) according to surface treatments: 1 (Primer): sandblasting with aluminum oxide particles 110 μm (Al2O3) + Alloy Primer (Kuraray); 2 (Cojet): sandblasting with silica oxide particles with Cojet-Sand + Silane ESPE-Sil. The conditioned blocks of each group were cemented, with Panavia F, to resin blocks under constant load of 750 g/10 min. The sets were cut to obtain 4 samples with dimensions of 10x1x1 mm per block (n = 10) and the adhesive surface with approximately 1 mm2. The microtensile test was done in the universal testing machine at 1 mm/min crosshead speed. The values of bond strength and standard deviation (MPa) were: Au P: 7.33 ± 1.93d; Au C: 13.35 ± 2.18c; NiCr P: 23.56 ± 6.5b; NiCr C: 42.6 ± 5.84a; Ti P: 26.17 ± 1.94b; Ti C: 44.30 ± 2.3a. Data were analyzed by variance test (ANOVA) and Tukey's test, $p < 0.05$. **Results:** The results indicated that the conditioning with treatment 2 increased the bond strength between the resin cement and alloys. The lowest bond strengths values were obtained with gold alloy, regardless the surface treatment. **Conclusion:** The results denied the hypothesis that the metallic alloys surface treatments do not alter the bond strengths values.

KEYWORDS

Resin cements; Dental materials; Dental Cements.

RESUMO

Objetivo: O estudo avaliou a influência de diferentes tratamentos de superfície em três ligas metálicas para próteses dentárias de metalocerâmicas (ouro; níquel-cromo; titanium), nos valores de resistência de união com cimento resinosos. **Material e Métodos:** Vinte blocos, 5 x 5 x 5 mm, de cada liga foram divididos em dois subgrupos (n = 10) de acordo com os tratamentos de superfície: 1: jateamento com partículas de óxido de alumínio (Al2O3) 110 μm + Alloy Primer (Kuraray); 2 (Cojet): jateamento com partículas de óxido de sílica com Cojet-Sand + Silano ESPE-Sil. Os blocos foram cimentados, com Panavia F, a blocos de resina sob carga constante de 750 g / 10 min. Os conjuntos foram cortados para obter assim, 4 amostras com dimensões de 10 x 1 x 1 mm por bloco (n = 10) e a superfície adesiva com cerca de 1 mm2. Foi realizado o teste de microtração na máquina de ensaio universal a 1 mm / min. Os valores de resistência de união e desvios -padrão (MPa) foram: Au P: 7,33 ± 1.93d; Au C: 13,35 ± 2.18c; NiCr P: 23,56 ± 6.5b; NICR C: 42,6 ± 5.84a; Ti P: 26,17 ± 1.94b; Ti C: 44,30 ± 2.3a, e analisados estatisticamente pelo teste de variância (ANOVA) e teste de Tukey, $p < 0,05$. **Resultados:** Os resultados indicaram que a resistência de união do condicionamento com o tratamento Cojet aumentou a resistência de união entre o cimento resinoso e ligas. **Conclusão:** Os valores mais baixos foram obtidos com liga de ouro, independente do tratamento de superfície.

PALAVRAS-CHAVE

Cimentos resinosos; Materiais dentários; Cimentos dentários.

INTRODUCTION

The retention of total or partial crown to the dental structure, which has been functioning as pillars of partial-fixed-dentures, is due to the mechanical principles created by geometry of the prosthetic tooth preparation performed by the dentist. Thus, employing luting agents as zinc phosphate cement, which does not present adhesive properties, is indicated without modifying the final clinical result. Performing prosthetic tooth preparations with more conservative dental reduction led to limited indication of adhesive fixed dentures with a metallic infrastructure due to the lack of adhesion between the luting agent and alloys composing these infrastructures.

The continuous technological development promotes the improvement of dental materials in order to create luting agents and alloys surface conditioners that significantly increase and maintain the adhesion between them. Besides that, there is an interest in developing resin cements mainly with new physical and chemical properties that presents greater resistance to degradation in oral environment [1,4].

Nevertheless, studies show that bond strength between the metal and luting agents, especially the resin ones, is increased when the metallic surface of prosthetic parts is submitted to specific surface treatments. Moreover, some authors have sought to develop alternative methods of surface treatment such as: sandblasting with oxides of different granulation, electrolytic attack, and conditioning with acid substances; with the aim at promoting higher and steady adhesive union values in oral environment [3,5-7].

One of these systems is Rocatec (ESPE, Seefeld - Germany), which uses initially a pre-treatment with aluminum oxide sandblasting (Rocatec-Pre), followed by a second sandblasting with silica oxide particles (Rocatec-Plus), finishing the conditioning with application of a silane layer on the sandblasted metallic surface (Rocatec-Sil). As for the system, the kinetic energy speed of the sandblasted silica particles is transformed into thermal energy that reaches the alloy fusion point between 1 and 2 μm from the surface. Consequently, the particles are joined to the alloy surface, forming a superficial layer of steady silica and with great chemical affinity to the silanization agents and resin materials [8-11].

According to the favorable adhesive properties created by the Rocatec System and the fact of being a system used mainly at prosthetic laboratories, a similar system called Cojet-Sand (ESPE, Seefeld - Germany) was introduced in the market, aiming to be used upon fractured metal-ceramic dentures infrastructures in mouth. This system also has the purpose to create silica covering on alloys, promoting higher union values with resin materials, though it differs from the Rocatec System because it uses a conventional sandblasting device at the dentists' offices and not a specific equipment of laboratorial usage. Repair techniques to metal-ceramic dentures with Cojet-Sand System were evident they provide higher adhesive union values between alloy surface and resin material than other types of surface conditioning [12-15]. Based on these results and the facility and versatility to use the Cojet-Sand, this system might be a viable alternative for surface conditioning of metal-ceramic denture alloys increasing the bond strength values to the resin cements.

The aim of this study was to evaluate the hypothesis that two surface treatments do not modify the bond strength values between resin cement and three metal-ceramic denture alloys.

MATERIAL AND METHODS

Wax blocks with dimension: 5 x 5 x 5 mm; were used as standard to cast twenty blocks in gold (Degudent U), titanium (Rematitan, Dentaurum) and nickel-chromium alloys (Wiron 99, Belgo). One of the surfaces of each metallic block was grounded following sandpaper on 300, 600, 800, 1000 and 1200 granulation in order to create a plain, smooth and uniform surface. Then, each one of the metallic blocks was duplicated with silicone (Express - 3M Dental Products, St. Paul, Mn - USA), obtaining a mold to make blocks of same dimension in microhybrid composite resin, which served as a base to enable the construction of the specimens. The specimens, blocks, were subdivided into two subgroups (n = 10) according to the applied surface treatment (Table 1):

Surface treatment 1: Adhesive monomer conditioner of metallic alloys – (Primer): Sandblasting with aluminum oxide particles (110 μm)/20 s, 2.8 bars, at an approximate distance of 10 mm perpendicular to the surface, followed by the application of an adhesive monomer layer (Alloy Primer, Kuraray CO., Japan).

Surface treatment 2: Silica oxide and silane (Cojet): With a micro sandblasting device (MicroetcherTM, Danville Engineering - USA), performinginitial sandblasting with aluminum oxide particles (110 μm) / 20 s / 2.8 bars, at an approximate distance of 10 mm perpendicular to the surface, followed by another sandblasting with silica oxide particles (30 μm), ending the conditioning with the application of a silane layer (Espe-Sil), waiting up 5 min for drying (Cojet-Sand System - ESPE, Seefeld - Germany).

Table 1 - Six experimental conditions

Experimental conditions	Alloys and conditions
G1 - Au p	Au-Pd Alloy sandblasted with aluminium oxide 110 µm and application of alloy primer.
G2 - Au C	Au-Pb Alloy conditioned with the Cojet-sand sistem and application of silane ESPE SIL.
G3 - NiCr p	NiCr Alloy sandblasted with aluminium oxide 110 µm and application of alloy primer.
G4 - NiCr C	NiCr Alloy conditioned with the Cojet-sand sistem and application of silane ESPE SIL.
G5 - Ti P	Ti Alloy sandblasted with aluminium oxide 110 µm and application of alloy primer.
G6 - Ti C	Ti Alloy conditioned with the Cojet-sand sistem and application of silane ESPE SIL.

After the conditioning procedures, the metallic blocks of each group were cemented to the composite resin blocks with resin cement (Panavia F. Kuraray CO, Japan), that was manipulated according to the manufacturer's instructions and applied to the surface of blocks. The specimen (metallic block / cement / resin block) was positioned, under load of 750 g perpendicular to the union surface, in a device that prevents any block movements that could alter the correct cementation procedure.

Before the cement setting, the excess was removed with an appropriate instrument, light cured (XL 3000 - 3M Dental Products, St. Paul, MN - USA), with light intensity of 450 mW / cm², 40 s on each side and posterior application of oxygen inhibiter (Oxyguard, Kuraray CO. - Japan) for 5 min on all the interfaces. Right after the recommended time of cementation, the sets were washed away with air-water spray and stored in distilled water at 37 ºC / 24 h.

For each group were obtained ten samples of metallic blocks cemented to resin blocks (Table 1). The cemented blocks were bonded with cyanoacrylate adhesive (Super-Bonder, Loctite, Piracicaba SP - Brazil) on a cylindrical acrylic base attached to a machine specially constructed to cut with carburundum discs with 0.15 mm of thickness and 22 mm of diameter, with a cut precision of 0.1 mm.

After the storage period, the samples had approximately 0.5 mm in its external faces cut off, and then generated slices with 10 x 5 x 1 mm (Figure 1).

Each slice was positioned and bonded to the 10 x 1 mm surface toward the metallic base, for further cutting to obtainthe specimens (Figure 1).

Thus, four samples were obtained, from each set of block, with the following characteristics: (a) rectangular shape, (b) quadrangular cross section (symmetric), (c) adhesive area of 1 ± 0.01 mm2 and (d) length of ± 10 mm (Figure 1).

Therefore, each group presented 40 means of bond strength values in MPa, being obtained for each block one mean value from the average of the four. Then, it was tested the statistical analysis based on ten means values for each group (n = 10).

The samples from each group were bonded with cyanoacrylate adhesive to a device for microtensile test, where they were positioned parallel to the axis of tensile load, minimizing possible lateral forces on the adhesive area. Each sample bonded to the device was submitted to tensile in a universal testing machine with load cell of 10 kgf (Model DL-1000, EMIC - Equipamentos e Sistemas Ltda., São José dos Pinhais - PR, Brazil) with crosshead speed of 1mm / min until the rupture (Figure 1).

The bond strength data were statistically analyzed by the analysis of variance test (ANOVA), being the block factor considered as a random effect and the alloy factor as impacted effect ($p < 0.05$).

Chemical Analysis

The analysis of the current chemical elements in metallic substrates was realized by Energy Dispersive Spectrometry (EDS) technique. Two samples from each experimental condition had the adhesive union surface

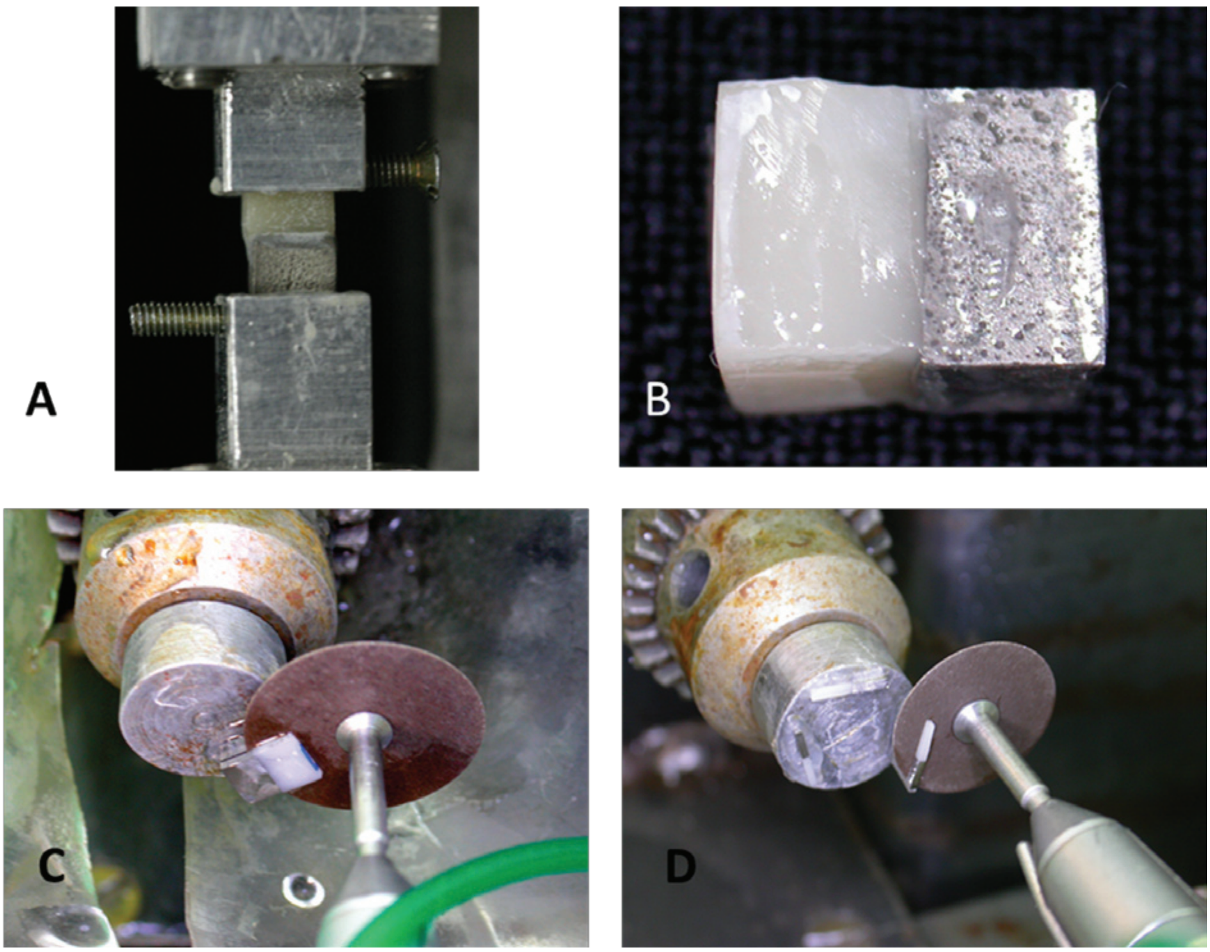

Figure 1 - A: fixation device; B: Zoom in of fixation device; C: Resin block cemented to the metallic block; D: cut of slices to obtain the specimens.

evaluated at EDS after the microtensile tests just to identify the current chemical element.

The x-ray microanalysis was realized upon the metallic alloys surface using: a scanning electron microscope (SEM) model LEO1450VP, from LEO-Zeiss, England; and an EDS system from Oxford with the program INCA Energy and EDS detector of resolution 133 eV. The measuring of each sample was made at a working distance of 15 mm and 20 kV acceleration voltage (20keV acceleration energy).

Thus, chemical composition analysis was done to obtain the concentration specters of each current chemical element in each one of the analysis samples (Figure 2).

RESULTS

The numerical summary-measures of central tendency (mean) and of dispersion (standard deviation, variance coefficient and values band) were calculated (Table 2).

Table 2 - Descriptive statistic of bond strength data (MPa)

Groups	Mean	sd	Var. Coef.(%)	
Au C CCC	10	13.35	2.18	16.39
Au P	10	7.33	1.93	26.38
NiCr C	10	42.60	5.84	13.71
NiCr P	10	23.57	6.50	27.56
Ti C	10	44.31	2.30	5.20
Ti P	10	26.17	1.94	7.42

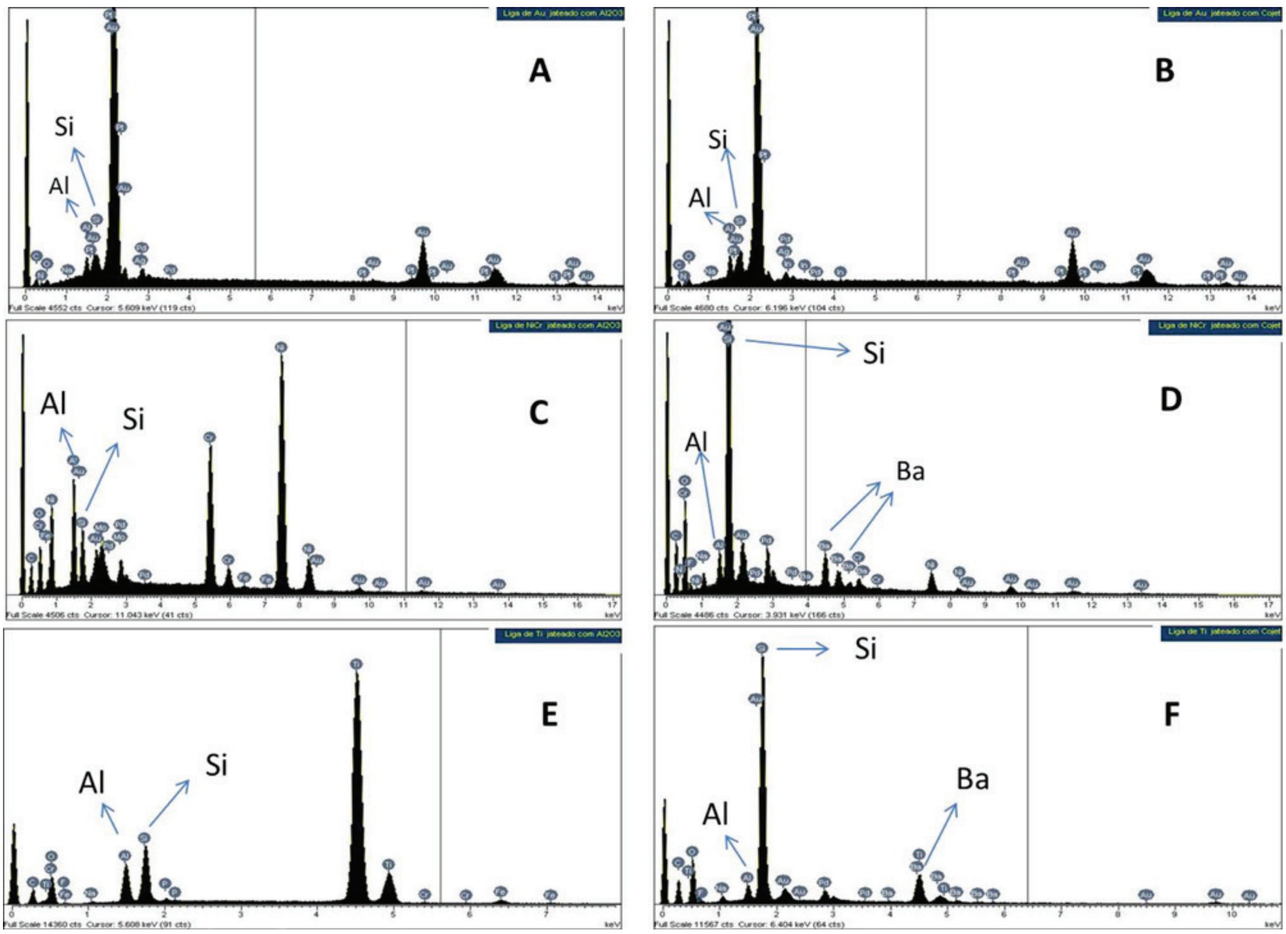

Figure 2 - EDS analysis of Ti, NiCr, Au alloy surface (sample) conditioned with two treatments, Cojet and Primer) after the microtensile test. A - Gold alloy/ treatment (Primer); B - Gold alloy/ treatment (Cojet); C- Nickel Chromium alloy/ treatment(primer);D- Nickel Chromium alloy/ treatment(Cojet); E-Titanium alloy/treatment (primer); F-Titanium alloy/treatment (Cojet).

Anova (Table 3) and Tukey's test (5%),revealed the nickel-chromium alloy presented the same performance as the titanium alloy, however with the gold alloy presented the lowest bond strength values.

Table 3 - ANOVA for the strength data obtained, with the microtensile test, in the randomized blocks experiment

Variation Source	gl	SQ	QM	F	P
Repetitions	3	113.1	37.7		
Blocks	9	626.8	69.6		
Residue I	27	1311.8	48.6		
Alloys	2	30442.6	15221.3	193.71	0.001*
Surface Treatment	1	12435.0	12435.0	158.25	0.001*
Residue II	195	15323.0	78.6		
Total	**239**	**62367.7**			

*P < 0.05

The Tukey's test performance, from the analysis of the data (mean) of Table 1, allowed grouping the six experimental conditions in four groups of similar performance, as indicated in Table 4.

Table 4 - Tukey's test (5%) homogeneous groups distribution

Experimental Condition		Mean	Homogeneous Groups
Alloy	Superficial Treatment		
Ti	Cojet	44.30	A
NiCr	Cojet	42.60	A
Ti	Primer	26.17	B
NiCr	Primer	23.56	B
Au	Cojet	13.35	C
Au	Primer	7.33	D

It was verified that the two experimental conditions with lowest bond strength values were represented by the gold alloy, mainly when held the conditioning with Primer. The experimental condition that produced the best bond strength performance was the one where the Cojet System was used as surface treatment. This condition differs from the others regardless the type of alloy used in.

Chemical Analysis (EDS) of Alloys Surface

The chemical analysis using EDS allowed the identification of the current chemical elements in the alloys adhesive surface after the microtensile test performance.

The Figure 2 shows the analysis results obtained right after the microtensile test performance for the gold, nickel-chromium and titanium alloys, respectively, conditioned with treatments 1 (Primer) and 2 (Cojet). Besides the chemical elements characteristic of each alloy, it was verified a high amount of Aluminum (Al) on the three alloys. After the conditioning with treatment 2 (Cojet), it was also observed a high amount of aluminum element, however with a significant increase in the amount of the chemical element Silicon (Si). Nevertheless, for the nickel-chromium and titanium alloys conditioned with treatment 2 (Cojet) the Figures 3 D and F show the presence of the chemical element Barium (Ba), indicative of the remaining resin material.

DISCUSSION

The improper use of superficial conditioning methods to dental alloys has been responsible for most of adhesive failures between the metal and resin cements interfaces. It was verified in the literature a great variety of superficial conditioning methods that used for altering the alloys surface - modifying their morphological characteristics through chemical substances such as acids, aggregating particles to its structure by sandblasting, or by electrolytic deposition of ions chemically more reactive, and thus, promoting a higher union chemical affinity between the alloys with the resin cements [6,16-21].

Adhesive cementation techniques are commonly used to ensure the durability of ceramic indirect restorations. The clinical success of this type of restoration depends on quality and durability of the adhesion between the union agent and the alloy. The quality of that adhesion depends on the adhesive mechanisms partially controlled by the surface treatment performed on the metallic surface before cementation, which promotes micromechanical retention with the substrate.

The dental alloys for metal-ceramic are classified as noble alloys, composed of precious metals like gold, and non-noble alloys, composed of non-precious metals like nickel, chromium and titanium. It was verified in the literature studies that they, the studies, endeavor to develop superficial conditioning for each type of alloy, in order to promote surfaces with better mechanical and chemical adhesion [16,17,19,20,22]. So far, it is verified for noble alloys that were not established a superficial conditioning protocol capable to promote an effective and stable adhesion. For non-noble alloys, such as nickel-chromium, authors have been able to obtain high and stable bond strength values [2,19,20,23]. That difference between the alloys is mainly due to the capacity to form oxidation layers on its surface.

Studies evaluated repair techniques for metal-ceramic dentures and it was also verified that the Cojet System provided higher adhesive union values between the noble dental alloys surface and the resin material rather than other types of surface conditionings. Therefore, this research was designed intended to evaluate the efficiency of the Cojet System, both for the noble alloys (gold) and for non-noble alloys (nickel-chromium and titanium). The use of treatment 1 (Primer) served as variable, because it followed the protocol established by the manufacturer [12-14].

Studies verified that the use of Alloy Primer increased the bond strength values between resin cements and noble alloys [17,24].

However, the results of this study differed from the standards obtained study mentioned above, on the other hand, they agreed with other study [25], where gold alloy also had the lowest bond strength values, revealing low conditioning efficiency with silica and the Alloy Primer on noble alloys. Nevertheless, the treatment 2 (Cojet) promoted higher bond strength values than treatment 1, independently of the alloy evaluated. For the nickel-chromium and titanium alloys conditioned with the treatment 2 (Cojet) elevated values were obtained. Thus, the results of this study are justified based on other studies [3,6,23,25]; which concluded that the low reactivity of noble alloys did not allow formation of oxidation layers, reducing the capacity of chemical union with the union agents and the resin cements.

Besides, the chemical analysis by EDS performed on a random sample of each experimental condition was extremely important in this study, because it generated conclusive information about the results of each experimental condition.

Figure 2 indicated the presence of chemical elements added to alloy surfaces after the surface conditioning and the mechanical assays, with similar results in the literture [2,5,12,13,15]. In Figures 2 D and F it is observed that after conditioning with treatment 2 (Cojet) for nickel-chromium and titanium alloys there was a significant increase in the quantity of the chemical element Silicon (Si) and the presence of the chemical element Barium (Ba). This fact suggested that cohesive failure actually occurred in the adhesive interface between resin cement and nickel-chromium and titanium alloys conditioned with treatment 2 (Cojet), because the chemical element Barium is a resin cement constituent and was not present on the surfaces of alloys conditioned with treatment 1 (Primer) that showed lower bond strength values.

CONCLUSION

The results denied the experimental hypothesis that surface treatments did not alter the bond strength values between resin cements and the surface of three alloys for infrastructure of metal-ceramic dentures. The results indicated that the conditioning with the treatment 2 (Cojet) increased the bond strength values to the three alloys evaluated. It was verified that the lowest bond strengths values were obtained with the gold alloy, independently of the type of surface conditioning.

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