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Surface change assessment of Co-Cr alloy subjected to immersion in denture cleansers

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Abstract

Choosing the right chemical cleanser for removable partial dentures is a challenge, because they present an acrylic and a metallic portion, which should be cleaned and not damaged. Aim: The aim of this study was to assess surface changes of cobalt chromium alloys immersed in different cleaners solutions: 0.05% sodium hypochlorite, 4.2% acetic acid, 0.05% sodium salicylate, sodium perborate (Corega Tabs®) and 0.2% peracetic acid. Material and Methods: One hundred and twenty circular specimens (10 mm in diameter) of two commercial available Co-Cr alloys were tested: GM 800 ® (Dentaurum) and Co-Cr® (DeguDent). The samples were randomly divided into ten experimental groups (n=10), according to the trend mark of alloy and cleaners solutions in which they were immersed, and two control groups, in which the samples of the two alloys were immersed in distilled water. Evaluations were performed through roughness measurement (rugosimeter Surftest 211, Mitutoyo), visual evaluation with stereomicroscope (Stereo Discovery 20, Carl Zeiss) and scanning electron microscope surface (JSM, 6360 SEM, JEOL), at experimental times T0 - before immersions, T1 - after one immersion, and T2 - after 90 immersions. Intergroup comparison for the effect of immersion in the different cleanser agents was evaluated through ANOVA/Tukey tests $(p \le 0.05)$. The effect of the time in the immersion of each alloy was evaluated by t-pared test $(p \le 0.05)$. The two alloys were compared using the t-Student test. **Results:** The analysis of roughness and microscopy showed that surface changes were significantly greater in groups submitted to 0.05% sodium hypochlorite after 90 immersions (T2). When comparing the two alloys, a similar behavior of roughness was observed for the cleaning agents. However, alloy GM 800® showed significant statistical difference for roughness variations in experimental times ($\Delta 1$ and $\Delta 2$), when immersed in sodium 0.05% hypochlorite. The number of exposures of the alloys to the cleaning agents showed a negative influence when using sodium hypochlorite solution. Conclusions: It is possible to conclude that 0.05% sodium hypochlorite has caused the greatest apparent damage to alloy surface.

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1. Introduction

Despite advances in materials and techniques in dental rehabilitation, removable partial dentures (RPD) remain as an important tool for public health, because they are a less costly option¹. Upon its installation in the oral cavity of patients, it is a dentist's

duty to instruct them about hygiene² to avoid the accumulation of biofilm, which is an etiological factor of oral diseases, such as caries and stomatitis. Patients can make use of mechanical and chemical cleaning methods. Their association has been reported in the literature as the best choice²⁻⁷, especially for special and geriatric patients, who find it difficult to properly brush their dentures¹. Techniques and materials should be effective in cleaning, and should not affect the components of the prosthesis.

Of the chemical cleansers used for full dentures, sodium hypochlorite solutions deserve special attention, since they degrade mucin and allow for greater removal of bacterial biofilm in depth^{2,8}. Solutions of sodium salicylate, sodium perborate, and acid peracetic are also used due to their antimicrobial potential⁸⁻¹⁰. A home-made option is vinegar (4.2% acetic acid) capable of reducing the number of bacteria on the surface¹¹.

Removable partial denture, however, present metallic components on their composition, normally cobalt chromium alloy¹². Choosing the right cleanser is a challenge, because solutions containing hypochlorites can cause some corrosion, staining, and even loss of physical properties^{13, 14}.

Thus, the aim of this study was to assess surface changes in cobalt chromium alloys subjected to immersion in different cleanser solutions: 0.05% sodium hypochlorite, 4.2% acetic acid, 0.05% sodium salicylate, sodium perborate (Corega Tabs®) and 0.2% peracetic acid.

2. Material and methods

2.1 Sample

One hundred and twenty circular specimens (10mm in diameter) of two different alloys of Cr-Co – Remanium GM 800 (Dentaurum, Pforzheim, Germany) and Co-Cr® Degudent (Dentsply Ind. and Co. Ltd, São Paulo, SP, Brazil) – were obtained through casting, polished with a sequence of wet sandpaper (600 to 2500) and diamond polishing paste with felt disk (Master Diamond Ferramentas, São Paulo, SP, Brazil) in polisher (Arotec APL-4, Arotec, Cotia, São Paulo, Brazil), numbered and randomly divided into 12 groups (n = 10). Ten experimental groups were designed, according to cleanser solutions – 0.05% sodium hypochlorite, 4.2% acetic acid, 0.05% sodium salicylate, sodium perborate (Corega Tabs®) and 0.2% peracetic acid; the type of alloy used (GM 800 ® or Co-Cr®); and two control groups, where the bodies and specimens were immersed in distilled water (control).

The alloys used have more than 85% of chromium and cobalt in their compositions: GM 800® (63.3% Co; 30% Cr; 5% Mo; 1% Si; 0.5% Mn; 0.4% C); CoCr® (64.8% Co; 28.5% Cr; 5.3% Mo; 0.5% Si; <1% Mn; <1% C; <1% N). Sample size calculation was performed for mean difference of roughness and it was found that at least seven samples should be used to achieve 80% power. Thus, 8 specimens were used for roughness assessment, and 2 were intended for microscopic analysis.

2.2 Immersion in cleansers

The cleanser solutions were prepared and 15 ml were poured into test tubes (Pyrex No. 9820, Corning Inc., USA), in which the specimens of each group were fully immersed.

For 0.05% sodium hypochlorite solution preparation, 5 ml

of 2.5% Sodium hypochlorite solution (Q-Boa®, Anhembi S/A Osasco, São Paulo, Brazil) was diluted in 200 ml of distilled water⁶. Immersion time for this solution was 10 min per cycle¹⁵.

The 4.2% acetic acid solution consisted of pure white vinegar (WMS Supermercados do Brasil S/A, Porto Alegre, RS, Brazil). Immersion time for this solution was 10 min¹⁶.

The 0.05% sodium salicylate solution was prepared by diluting 1/4 teaspoon (0.25g) of sodium salicylate PA (C7H5NaO3) (Vetec Química Fina LTDA, Rio de Janeiro, RJ, Brazil) in 250 ml of distilled water. Immersion time for this solution was 15 minutes².

Effervescent sodium perborate solution was prepared by diluting one tablet of Corega Tabs® (Stafford-Miller Ind., Rio de Janeiro, RJ, Brazil) in 150 ml of water at 45°C, as recommended by the manufacturer. Its immersion time was 15 minutes¹⁷.

The 0.2% peracetic acid solution was prepared by diluting 13.4 ml of 15% peracetic acid (Sigmasul, Cachoeirinha, RS, Brazil) in one liter of distilled water. Immersion time for this solution was 15 minutes.

Surface assessments were held before immersions (T0), after one immersion (T1), and after 90 immersion cycles (T2), simulating the daily use of these solutions for three months. The samples were cleaned with spray of distilled water and dried on absorbent sheet, between each immersion, and the interval between consecutive immersions were merely the time necessary to wash and dry the specimens.

2.3 Surface Roughness

Surface roughness was measured using a rugosimeter (Surface roughness was measured using a rugosimeter (Surftest SJ 211, Mitutoyo Corp., Kanagawa, Japan), with 6 readings with cut-off of 0.25 mm in each specimen, 3 on the x-axis (x) and 3 on the ordinate axis (y). The Ra parameter, which provides the means of peaks and valleys, was assessed in all experimental time intervals (T0, T1, and T2), using the center of the sample.

2.4 Microscopic assessment

Two microscopic analyses were performed. Initially, a stereomicroscope was used with magnification of 8.5x to assess samples in 3 experimental times. A damage index was created for the surface, where 0 indicates the absence of any signs of changes; 1, the loss of brightness and light surface deposition; 2, the occurrence of spots in more than two thirds of the surface of the specimens; and 3, the total darkening of the specimens. After that, Scanning Electron Microscopy (SEM) was performed with magnification of 500x, in order to view the topographic surface appearance of alloys. With the use of X-ray energy dispersive spectroscopy (EDS), it was possible to determine which chemical elements were present on the surface.

2.5 Statistical analysis

Data were tabulated and statistically analyzed using SPSS (Statistical Package for Social Sciences, version 13.0). Normality was verified by the Shapiro-Wilk test. Surface roughness after the application of cleaning protocols at times T1 and T2, as well as the difference found by subtracting roughness after immersion (in both

times) by the initial roughness (baseline), were compared between different experimental groups by analysis of variance and multiple comparison Tukey test ($p \le 0.05$). Roughness data after immersion in cleanser solutions were compared to initial (baseline) by paired t test ($p \le 0.05$). The two alloys were compared with respect to roughness in the various protocols through t-Student test ($p \le 0.05$).

The captured images of sample surface changes were visually assessed twice in an optical stereomicroscope by one observer, to yield a Kappa coefficient of 0.87. The scores to visual changes underwent transformation "rank" to then be compared between the different experimental groups by analysis of variance and multiple comparison test of Tukey ($p \le 0.05$). The two alloys were compared in scores of visual changes on different protocols by Mann-Whitney test ($p \le 0.05$).

3. Results

3.1 Surface Roughness

Results showed no statistically significant difference between the methods of cleaning after the first immersion as to roughness (T1). After 90 immersions (T2), the means of Ra (μ m) in the groups submitted to 0.05% sodium hypochlorite were significantly higher (Table 1). Other cleansers did not cause surface roughness changes in the alloy over time (Table 1). When comparing the two alloys, we have found similar behavior in roughness for cleansers. However, alloy GM 800® showed significant statistical difference between Δ 1 and Δ 2 when immersed in 0.05% sodium hypochlorite (Table 2).

Table 1 - Mean and standard deviation (SD) of surface roughness Ra (µm) of alloys immersed in various cleansers in experimental times

Alloy	Solution	Ra (µm) - T0	Ra (µm) - T1	Ra (µm) - T2
Co-Cr®	0.05% sodium hypochlorite	0.050 (0.009) ^{aA}	0.063 (0.011) ^{aB}	0.494 (0.083) ^{bC}
	4.2% acetic acid	0.061 (0.017) ^{aA}	0.070 (0.027) ^{aA}	0.059 (0.021) ^{aA}
	0.05% sodium salicylate	0.061 (0.016) ^{aA}	0.068 (0.035) ^{aA}	0.083 (0.037) ^{aA}
	Sodium perborate (Corega Tabs®)	0.047 (0.006) ^{aA}	0.060 (0.019) ^{aB}	0.067 (0.010) ^{aB}
	0.2% peracetic acid	0.052 (0.013) ^{aA}	0.067 (0.029) ^{aAB}	0.080 (0.032) ^{aB}
	Distilled water - control	0.062 (0.021) ^{aA}	0.069 (0.018) ^{aA}	0.060 (0.011) ^{aA}
GM 800®	0.05% sodium hypochlorite	0.053 (0.015) ^{aA}	0.077 (0.021) ^{aB}	1.254 (0.191) ^{bC}
	4.2% acetic acid	0.073 (0.019) ^{aA}	0.088 (0.023) ^{aA}	0.103 (0.048) ^{aA}
	0.05% sodium salicylate	0.062 (0.026) ^{aA}	0.068 (0.026) ^{aA}	0.072 (0.027) ^{aA}
	Sodium perborate (Corega Tabs®)	0.062 (0.017) ^{aA}	0.074 (0.021) ^{aB}	0.074 (0.018) ^{aAB}
	0.2% peracetic acid	0.085 (0.034) ^{aA}	0.099 (0.042) ^{aA}	0.084 (0.032) ^{aA}
	Distilled water - control	0.072 (0.023) ^{aA}	0.087 (0.038) ^{aA}	0.075 (0.033)ª ^A

Different lowercase letters indicate statistically significant difference between the immersion solutions, with the same alloy and time (ANOVA/Tukey, $p \le 0.05$).

Different capital letters indicate statistically significant difference among immersion times (t-pared test, $p \le 0.05$).

Table 2 - Mean and standard deviation (SD) of the variation of roughness after 1 (Δ 1) and 90 immersions (Δ 2).

Solutions		Co-Cr®	GM 800®
0.05% adjum hypophlarita	Δ1	0.013 (0.012)ª	0.024 (0.017)ª
0.05% sodium hypochlorite	Δ2	0.446 (0.085)ª	1.202 (0.194) ^b
4.2% acetic acid	Δ1	0.008 (0.030)ª	0.014 (0.024)ª
	Δ2	0.000 (0.028)ª	0.030 (0.042)ª
0.05% sodium salicylate	Δ1	0.007 (0.041)ª	0.007 (0.034)ª
0.05% Soulum Salicylate	Δ2	0.021 (0.037)ª	0.010 (0.021)ª
sodium perborate (Corega Tabs®)	Δ1	0.014 (0.017)ª	0.010 (0.013)ª
souluin perporate (Corega Tabs®)	Δ2	0.022 (0.014) ^a	0.010 (0.029)ª
0.2% peracetic acid	Δ1	0.013 (0.025)ª	0.015 (0.045)ª
	$\Delta 2$	0.028 (0.034)ª	0.000 (0.037)ª
distilled water - control	Δ1	0.002 (0.021)ª	0.003 (0.020)ª
	Δ2	0.071 (0.030)ª	0.083 (0.036)ª

Different letters indicate statistically significant differences among alloys for roughness variation $\Delta 1$ and $\Delta 2$ with the same cleansers (p ≤ 0.05).

3.2 Microscopic Assessment

After one immersion (T1) no clear visual change was noted on the surface of any of the groups. However, after 90 immersions (T2), the two alloys submitted to 0.05% sodium hypochlorite presented the highest scores, pointing to the major changes (Table 3).

Table 3 - Mode c	f visual	l scores aft	er 90 in	mmersions ((T2).
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Solutions	Co-Cr®	GM 800®		
0.05% sodium hypochlorite	2ª A	3ª B		
4.2% acetic acid	0 ^{b B}	0 ^{с в}		
0.05% sodium salicylate	1 ^{b B}	1 ^{bB}		
Sodium perborate (Corega Tabs®)	0 ^{b B}	0 ^{c B}		
0.2% peracetic acid	0 ^{b B}	1 ^{b C}		
Distilled water - control	1 ^{b B}	0 ^{c B}		

Different lowercase letters indicate statistically significant difference between the columns (difference between the alloys - Mann-Whitney test, $\alpha = 0.05$). Different capital letters indicate statistically significant difference between the lines (difference between immersion solution - ANOVA/Tukey, $\alpha = 0.05$).

In further analysis by SEM, it was possible to observe sharpening occurrence, suggesting a slight texturing of the surface of the Co-Cr® alloy after the first immersion. However, after the ninetieth immersion, widespread surface change was noticed, with the presence of protruding clusters and occasional depressions (Figure 1). For the GM 800® alloy, after the first immersion, minimal superficial change was noticed. However, after the ninetieth immersion, the image shows more abrupt changes in the structure of the sample (Figure 2). EDS surface analysis of both alloys, when immersed in 0.05% sodium hypochlorite, showed the presence of oxygen and chlorine, which indicates corrosion. Iron and tungsten were also found in the composition of alloy GM 800®, not reported by the manufacturer.

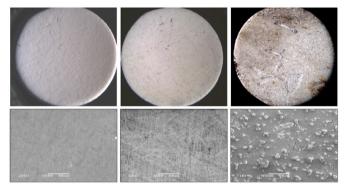


Fig. 1 - Co-Cr \mathbb{R} alloy specimen immersed in 0.05% sodium hypochlorite solution in the three experimental times (a: T0; b: T1; c: T2). Above there is the stereomicroscope image (Magnification of 8.5x). Below, SEM images (magnification of 500x). T1 shows little change in surface brightness (score 1), while T2 shows a widespread staining on the surface (score 2).

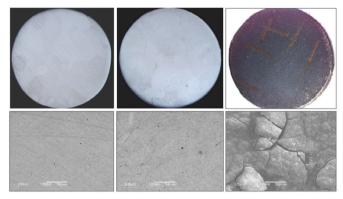


Fig. 2 - GM 800 alloy specimen immersed in 0.05% sodium hypochlorite solution in all three experimental days (a: T0; b: T1; c: T2). Above there is the stereomicroscope image (magnification 8.5x). Below, SEM images (magnification 500x). T1 shows little change in the surface brightness (score 1), while T2 is observed to such darkening the surface and abrupt changes in relief.

4. Discussion

When choosing a metal for facing different challenges in hostile environments, its corrosion behavior its corrosion behavior is the most important factor to be considered¹⁸. Thus, this work has compared the Co-Cr® e GM 800® metal cobalt chromium alloys, after being immersed in 5 cleaners.

Regarding cleanser comparison, 0.05% sodium hypochlorite solution caused more obvious changes to alloy, generating higher roughness values and higher scores on the analysis with stereomicroscope. Analysis of roughness after immersion of the alloy in 4.2% acetic acid solution, 0.05% sodium salicylate, sodium perborate (Corega Tabs®) and 0.2% peracetic acid, showed no statistically significant difference between experimental periods with no increased roughness over time. It was also observed that water-immersed alloys (control) had scores of 0 and 1, with slight loss of brightness (score 1) only seen with a microscope and not to the naked eye. Therefore, we decided to consider these two as having no damage. Thus, 4.2% acetic acid solution, 0.05% sodium salicylate, sodium perborate (Corega Tabs®), and 0.2% peracetic acid did not cause visible damage to alloys at different experimental times.

When the alloys were compared, it was observed that, with regard to roughness, the nominal values of Ra (µm) were higher for GM 800®, but with no statistically significant difference. However, statistically significant difference has been found for $\Delta 1$ and $\Delta 2$ for this alloy. With the stereomicroscope, clearer changes were observed for alloy GM 800® after 90 immersions (T2). The evaluation by SEM confirmed most surface changes for this alloy at T2. As for the CoCr® alloy, it showed superficial changes similar to those that occur when superficial electrochemical attack is conducted with acid solution to metallographic analysis¹⁹, with the view of protruding beads on the surface of the alloy. GM 800® alloy showed greater degree of change with suggestive image of detachment of surface oxidation plates. It is believed that the observed difference for the two alloys at T2 may be related to the fact that GM 800® has shown iron and tungsten in its composition, which was identified by EDS, since the presence of other metals in the alloy can modify its corrosion resistance and increase the speed of etching²⁰.

It is believed that surface roughness of the alloy reaches clinical significance when reaching 0.2 µm, which favors the adhesion of biofilm. Thus, values higher than this cannot be clinically accepted²¹. In this study, the two alloys exceeded this cut-off point after 90 immersion in 0.05% sodium hypochlorite (Co-Cr \mathbb{R} = 0.446 μ m; GM 800 \mathbb{R} = 1.202 μ m), which suggests that 0.05% sodium hypochlorite may cause damage the Co-Cr alloys used in RPD, which agrees with the literature^{2,13,14,22}. Although sodium hypochlorite has fungicidal^{8,16,23} and bactericide effect, and is able to penetrate up to 3 mm in the resin, not only eliminating the surface bacteria, but also those in depth, if allowed to act for ten minutes, at a concentration of 0.525%¹⁵, its use in RPD should be cautious due to the deleterious effects on metal infrastructure. Recent studies have demonstrated the damaging effect of sodium hypochlorite on the alloy Co-Cr by weight and ion loss²², and by reducing the modulus of elasticity and ultimate strength. In the latter study, however, the property of bending was found satisfactory according to ADA specification No.14²⁴.

With respect to the quantity of infused over time, only groups exposed to sodium hypochlorite 0.05% showed obvious changes after the first immersion. Comparing evaluations in SEM's first exposure to hypochlorite (T1) with the evaluation after 90 exposures (T2), it is clear that there was a real deterioration of the surface of the two alloys, which is higher in the alloy (GM

800 R). The visual scores evaluation showed scores 2 and 3 after dipping 90 cycles, while after the first immersion the score was 0, agreeing with results of previous studies^{25, 26}.

With the exception of the groups submitted to 0.05% sodium hypochlorite solution, there was no occurrence of surface damage to the alloy. Therefore, it is possible to perform removable partial denture cleaning with most solutions used in the study. However, further studies are needed for evaluating the mechanical properties of alloys, as well as evaluate more immersions.

5. Conclusion

The solution of 0.05% sodium hypochlorite showed significant surface changes, suggestive corrosion, while other solutions did not present such deleterious effects. Both alloys showed similar surface changes after 90 immersion cycles for different cleansers. Increased contact with cleansers caused greater surface changes on the alloy only when 0.05% sodium hypochlorite solution was used.

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