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ORIGINAL ARTICLE

Surface characterization of passive film and elemental release analysis of a Ni–Cr alloy during bleaching, part I: effects of different bleaching agents

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Abstract The aim of this study was to evaluate the elemental release from a Ni–Cr dental casting alloy subjected to 10% hydrogen peroxide (HP) or 10% carbamide peroxide (CP) solutions and to determine the composition of surface oxide layer formed on alloy samples. Cylinder-shaped 15 specimens were cast from a Ni–Cr alloy and divided into three groups ($n=5$). Samples were exposed either to phosphate-buffer solution, HP, or CP for 30 days, and total mass and individual elements (Ni, Cr, Mo) released into solutions were measured by means of atomic absorption spectrometry. Before and after elemental release measurements, a scanning electron microscope (SEM) accompanied by energy dispersive spectroscopy (EDS) (SEM/EDS) was used to analyze the surface morphology, and surface characterization of passive film formed on alloy samples was also performed by using X-ray photoelectron spectroscopy (XPS). The presence of bleaching agents induced the mass released compared to control group (4.9 $\mu g/cm^2$); this effect was recorded in both HP (171.2 $\mu g/cm^2$) and CP (59.7 $\mu g/cm^2$). XPS data showed that Cr and Ni levels in oxide layers formed on HP group were higher, Mo level was lower than those of CP group.

Keywords Bleaching · Biocompatibility · Elemental release · Ni–Cr alloys · Surface oxide layer · XPS

Introduction

Nickel-based alloys are commonly used for many prosthodontic applications [1]. When selecting a dental casting alloy for a specific clinical situation, three factors influence the clinician′s decision: cost, physical properties, and biocompatibility [2]. The corrosion behavior of dental alloy is of great interest as an indication of their biocompatibility [3] because these alloys are in long-term intimate contact with oral tissues [4]. Corrosion may be described as the deterioration of materials by aggressive action of the environment. The oral environment is very conducive to corrosion. Fluctuations in temperature, the presence of moisture, changes in pH because of diet, variations in oxygen pressure, and decomposition of food all contribute to this process [5]. Corrosion of alloys occurs when elements in the alloy ionize [6]. The biocompatibility of an alloy depends on the type of an alloy and the elements released from it into the surrounding medium or tissues [7]. Released metallic ions are critical with respect to the biological behavior of dental metals and alloys [8]. Corrosion can be measured either by many forms of electrochemical tests that measure elemental release indirectly through the flow of the released electrons or by tests that measure the release of the elements directly by spectroscopic methods [6]. Both methods are commonly used, but elemental release is probably more relevant to any biological effects that corrosion might have [6, 9].

Corrosion resistance of Ni-based alloy depends greatly on the formation of a thin, protective oxide film (passive film) on the surface of the material [10]. Chromium is the main alloying element in Ni-based alloys and is added to improve the alloy′s ability to form a protective oxide film on the surface [1, 5, 10]. It has been suggested that the addition of Mo will further enhance the corrosion resistance of the nickel-based alloys [5, 10]. Surface analysis of the

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passive oxide film formed on the surface of Ni-based alloys has related increased corrosion rates to lower proportions of Cr and Mo in the film [11, 12].

When a dental alloy is embedded in the acidic oral cavity, the corrosion resistance of passive film formed on the alloy should be of great concern due to the biological considerations. The proper characterization of passive film is the first step in evaluating biocompatibility. If the passive film, normally in the form of oxides, of the dental alloy is damaged, increased metal ions and/or corrosion products may be released to adjacent and systemic tissues [13].

Mouthguard bleaching is a popular method for treating tooth discolorations [14]. For brightening discolored teeth, hydrogen peroxide (HP) or peroxide releasing agents, such as sodium perborate and carbamide peroxide (CP) are generally used [15, 16]. The ability of these products to lighten tooth color is evident, but the safety of some of these oxidizing agents is a subject of concern [16].

Most mouthguard bleaching procedures are considered relatively safe with regard to systemic effects. Since Ni–Cr dental casting alloy is the most widely used restorative material for premolars and molars, patients undergoing mouthguard bleaching are likely to have crown-restored teeth. Although bleaching materials are usually applied to the anterior teeth, excessive bleaching agent may make contact with metal restorations and enhance element release from these restorations [14, 17]. The study including mercury release from dental amalgam exposed to bleaching agent revealed that this procedure causes mercury release in solution [14]. Canay et al. [18] investigated the effect of 10% carbamide peroxide on the electrochemical corrosion of dental casting alloys used for fixed partial dentures and amalgam. Their results revealed that unpolished amalgam and Ni–Cr alloy samples had the most corrosion rates, and noble alloys had the least corrosion rates. Ameer et al. [19] investigated electrochemical behavior of a non-precious dental alloy in bleaching agents and concluded that all tested agents may affect its corrosion behavior.

Several studies have involved in vitro elemental release analysis of dental alloys under different test conditions [2, 20–25]. Nelson et al. [2] conditioned dental casting alloys in either saline, cell-culture medium, or a saline/bovine serum albumin solution; Messer and Lucas [20] used a cell-culture medium supplemented with 5% fetal bovine serum before measuring elemental release. Geis-Gerstorfer et al. [21] studied elemental release from dental casting alloys in 0.1 mol/L lactic acid and 0.1 mol/L sodium chloride solution. Consideration of published studied shows that relatively little attention has been directed at understanding the mechanisms responsible for metal ion release from dental alloy following exposure to bleaching agents.

Elements released from dental alloy during mouthguard bleaching can be absorbed by the oral mucosa as well as by the respiratory and gastrointestinal tracts [17]. Therefore, it is of clinical importance to assess the amounts of released elements from dental alloy exposed to commonly used bleaching agents. In addition, surface alterations of dental alloy related with bleaching agents should be determined. The aim of this study was to evaluate the elemental release from a Ni–Cr dental casting alloy subjected to 10% HP and 10% CP solutions and to determine the composition of surface oxide layer formed on alloy samples.

Materials and methods

The materials used in this study are 10% HP solution, 10% CP solution, phosphate buffer solution (PBS) at pH 6.5, and a Ni–Cr dental casting alloy (Wiron 99®, BEGO, Bremen, Germany). The bulk composition for the alloy is listed in Table 1. Cylinder-shaped (4 mm in diameter×10 mm in height) 15 samples were cast using an induction casting machine (Ducatron Serie 3, Ugin'dentaire, Seyyins, France). Polishing procedure was performed until obtaining clinically acceptable surfaces in accordance with the recommendations of the manufacturer. Briefly, after devesting, the alloys were blasted with 250 µm Al_2O_3 and finished with diamond milling tools. Next, the surfaces were blasted with 50 µm Al_2O_3, then blasted-polished with Perlablast® (BEGO, Bremen, Germany), rubber-polished with rubber polishers, finish-polished with a polishing paste and steam cleaned.

Finally, each sample was cleaned by scrubbing with a soft toothbrush and 2% glutaraldehyde solution, rinsing with distilled water, and ultrasonic treating in 70% isopropyl alcohol. After cleaning, samples were disinfected in 70% isopropyl alcohol for 20 min, followed by two rinses with sterile double-distilled water to remove the alcohol [23, 24]. After that, the alloy samples were randomly divided into the test and control groups with five samples each.

The testing samples were each sealed in individual assay test tubes containing 2.5 mL solution of either 10% HP or 10% CP at pH 6.5. The 10% HP solution was prepared by diluting the 30% HP solution in 0.1 M PBS, and the 10% CP solution was prepared by dissolving carbamide peroxide powder (Sigma, St Louis, MO, USA) in 0.1 M PBS, adjusting the pH accordingly. The remaining samples were sealed in similar assay test tubes containing 2.5 mL 0.1 M PBS at pH 6.5 which served as controls. Negative controls for the extraction experiments were the extracting solutions without alloy samples. The assay tubes containing control samples as well as those without samples were incubated at 37°C in a 95% air, 5% CO_2 atmosphere for 30 days. Distinctly from the control group, samples in the testing groups were exposed to fresh HP and CP solutions only 8 h per day, and the solutions were collected following their application; after completion of 8 h, samples were rinsed with sterile double-distilled water to remove the bleaching agents, then transferred into assay tubes containing 2.5 mL 0.1 M PBS for the rest 16 h per day within this 30 days time period. Testing groups were also kept in incubator during whole time interval. All the extracting solutions were renewed everyday to avoid microbial overgrowth and drift of the pH of the medium [2]. Since the total surface area of each sample was approximately 1.5 cm^2, the ratio of sample surface area to the volume of the extracting solution was approximately 0.6 cm^2/mL, which was in the limits (0.5 to 6.0 cm^2/mL) of that required by the International Standards Organization for testing of this type [26]. Elements released (total mass and individual elements) into solutions were measured by graphite furnace atomic absorption system using Varian Atomic Absorption Spectrophotometer (30/40 model, Varian Techtron Pty Ltd., Victoria, Australia) during a 30-day period. Each measurement was repeated five times for each sample and the mean values were calculated. Values for the concentrations obtained from the atomic absorption measurements were converted to micrograms of mass released from the alloy per square centimeter of alloy surface for each element.

One-way analysis of variance was applied to compare the mass loss among the three groups. When differences were detected, Dunnett T3 multiple range comparison tests (α=0.05) were used to identify statistical differences between the groups within a specific condition.

Before and after elemental release measurements, a scanning electron microscope (SEM, Jeol JSM-6400, Tokyo, Japan) accompanied by an energy dispersive spectroscopy (EDS, Thermo Noran, NORAN System Six-NSS300, Middleton, USA) facility (SEM/EDS) was used to analyze the surface morphology (two samples of each group). Prior to casting, the ingot were examined with EDS, and an ingot was also examined using X-ray fluorescence spectroscopy (XRF, RIX 3000, Rigaku Industrial Co., Yamaguchi City, Japan) in order to determine the composition of the alloy.

In addition, before and after bleaching applications, surface characterization of passive film formed on alloy samples was also investigated by using X-ray photoelectron spectroscopy (XPS, Specs ESCA, Berlin, Germany). One cylinder from each group was analyzed with XPS employing a Mg anode with Kα radiation=1253.6 eV. The analysis area on each sample was a circle (approximately 4×7 mm). The binding energies (BE) for all photopeaks were corrected by assigning the C 1*s* peak due to surface contamination at 285.0 eV. XPS spectra were recorded with a step size of 0.1 eV and a scan number of 5; dwell time for each energy step was 1 s. The atomic percent of each element on the surface area was calculated from the photopeak area using a software (SpecsLab software). To obtain composition as a function of depth (depth profiling), the surface of alloy was etched using argon sputtering with an ion beam for times between 0 and 3,480 s with 120 s intervals.

Results

Table 1 shows the EDS and XRF ingot compositional analyses of the Wiron 99® (W99) alloy compared with the manufacturer's information. The results are very similar for the major elements, with the only differences being the minor elements, identified by EDS and XRF, but which the manufacturers do not always quote.

The release of elements from the groups subjected to different extracting solutions was examined over a period of 30 days; afterwhich, the total mass and individual elements released from all samples were different. The amounts of mass released, as determined through elemental release measurements, are listed in Table 2.

During immersing into PBS, samples were almost stable, releasing only minor amounts (4.9 μg/cm^2) of elements. Elemental release was significantly elevated with two bleaching solutions compared to control group (Table 2). The increases in elemental release were highest for the HP solution (171.2 μg/cm^2), which was approximately three times greater than those of CP solution (59.7 μg/cm^2). In all cases, the release of elements was statistically different from each other (α=0.05), and the order of elements released was as follows: Ni>Mo>Cr.

Table 1 The bulk composition of Wiron 99® as supplied by the manufacturer, from EDS and XRF analyses

Elements	EDS (at.%)	XRF (mass%)	Manufacturer (mass%)	Manufacturer (at.%)
Ni	63.50	61.7	65.0	65.2
Cr	24.85	22.1	22.5	25.5
Mo	8.93	8.6	9.5	6
Si	2.72	2	1.0	2
Nb	ND	0.9	1.0	0.6
Fe	ND	0.2	0.5	0.5
Ce	ND	ND	0.5	0.2

n=2

ND not detected

Table 2 Summary of AAS data (µg/cm^2)

	PBS (control)	HP	CP
Cr[a]	0.3 (0.02)	27 (1.67)	7.1 (0.18)
Ni[a]	2.9 (0.52)	77.4 (16.22)	30.4 (1.03)
Mo[a]	1.7 (0.16)	66.8 (15.89)	22.2 (2.71)
Total[a]	4.9 (0.2)	171.2 (13.31)	59.7 (1.30)

SD in parentheses

[a] Results are the arithmetic mean of five analyses of each five samples

The post-corrosion examination with SEM revealed that both HP and CP solutions caused little surface erosion compared to untreated samples (Fig. 1), but the surface erosion that occurred with HP solution was more prominent with respect to CP solution.

Results of XPS analysis of surface oxide layer formed on Ni-based alloy samples before and after bleaching applications are listed in Tables 3 and 4, showing the compound structure and composition content (in atomic percent), respectively. Representative XPS survey scans are shown in Fig. 2a, b; XPS depth profile analysis of the Ni–Cr alloy are shown in Fig. 3. Ni, Cr, Mo, and O were shown to be present on the surfaces of all tested samples. Surfaces were a mixture of Cr, Mo, and Ni oxides for all groups. The presence of carbon was due to organic species adsorbed on the high energy oxide surface.

For control group (untreated sample), a complex Ni 2*p* peak was present on the surface with BE that correlated with metallic Ni (Ni^0) and NiO. A complex asymmetric Cr 2*p* peak, indicating the presence of Cr_2O_3 as well as metallic Cr (Cr^0), was also identified. Surface exhibited a complex Mo 3*d* peak composed of metallic Mo (Mo^0) and MoO_3 (Tables 3 and 4). Depth profiling analysis showed that oxide forms of Ni, Cr, and Mo were detected for only argon sputtering times until 1,200–1,440 s. After this time, there was no evidence about their oxide forms. O accounted for 26.4 at.% of the oxide layer. The minor alloy elements (Si, Nb, Ce, Fe) were not detected.

For the CP-treated group, NiO was present for only argon sputtering times between 0–120 s; after this time, Ni was detected only in elemental state. Mo was present in both oxide and elemental states. There was no evidence of Cr, Ce, and Nb in the elemental state, but only in the oxide forms. O accounted for 62.4 at.% of the oxide layer. Trace amounts of Ce, Fe, and Nb were also detected. Si (6.2 at.%) was detected only in the oxide form. The concentrations of Ni and Cr were reduced, while the concentrations of all other elements detected were increased (especially O and Mo) compared to untreated group.

For HP-treated group, in addition to oxide forms shown in Table 3, binding energies that correlated with Ni_2O_3, Mo_2O_5, CrO_3, and NbO_2 were also present. Trace amounts of Fe and Nb were also detected. Different from the other two groups, significant amount of Ce (8.3 at.%) was demonstrated. There was no evidence of Cr, Ce, and Nb in the elemental state, but only in the oxide forms. Ni and Mo were present in both oxide and elemental states. Si (5.7 at.%) was detected only in the oxide form. O

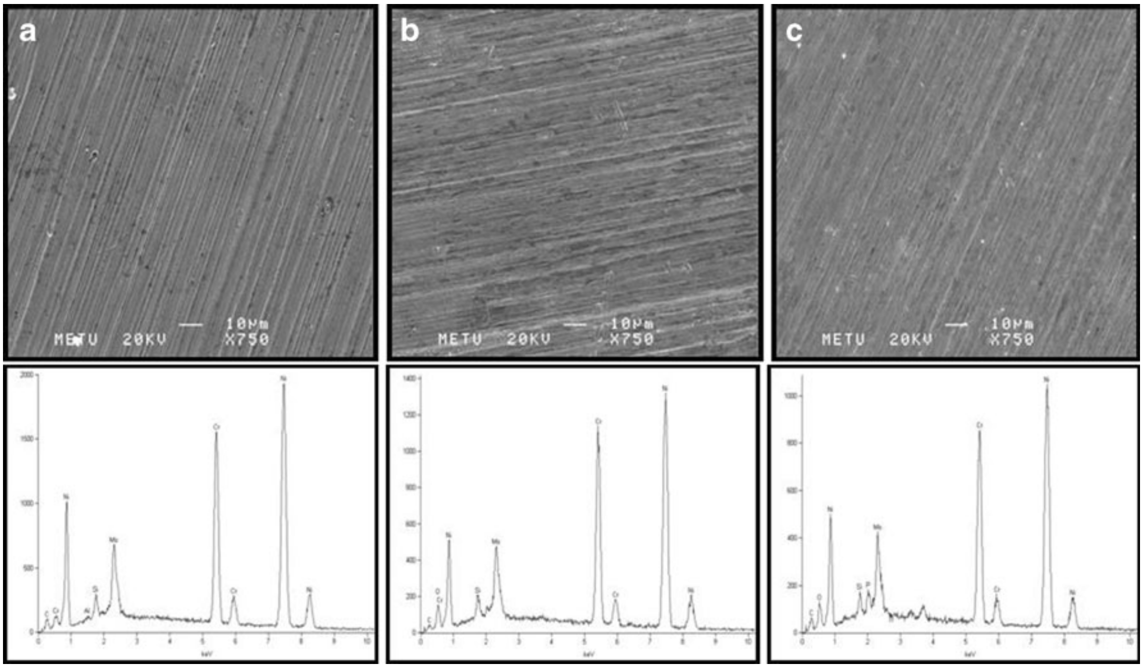

Fig. 1 SEM micrographs and corresponding EDS element profiles for W99 alloy comparing before (**a**) and after corrosion measurements [HP treated samples (**b**), and CP treated samples (**c**)]

Table 3 Results of XPS chemical analysis of surface passive film before and after elemental release measurements, showing the compound structure

	NiO	Cr_2O_3	MoO_3	SiO_2	Nb_2O_5	CeO_2	Fe_2O_3
Before immersion	+	+	+	–	–	–	–
HP immersion	+	+	+	+	–	+	+
CP immersion	+	+	+	+	+	+	+

accounted for 66.8 at.% of the oxide layer. Except for Ni, the concentrations of all elements detected were increased compared to untreated group. Mo, Si, and Nb levels were decreased, while the all other elements were increased compared to CP-treated group (Table 4).

Discussion

In this study, the corrosion behavior of the W99 alloy investigated was due to the elemental release in bleaching solutions. The results showed that bleaching treatment caused an increase in elemental release from Ni-based alloy. In addition, bleaching agents caused alterations to the chemistry and the surface morphology of this alloy.

It has been shown that a lower corrosion resistance occurred for Ni-based alloy with a lower Cr content (12.6 wt%) compared with a higher Cr content (25 wt%) in the bulk alloy [27]. Huang [11] also showed a lower corrosion resistance for alloys with 12.4–13.6 wt% Cr compared with alloys containing more than 21 wt% Cr that were resistant to corrosion. In the current study, tested alloy with 22.5 wt% Cr in the bulk surpassed the mentioned level for corrosion resistance (Table 1). In considering the choice of alloy applied in this study, the selection of a low corrosion resistant alloy in addition to W99 could provide a better understanding for making comparisons, but the experimental design in the current study was not in excess of what is anticipated under normal use (conditions). Testing a high-corrosion resistant alloy can also be used as further confirmation of the safety of these types of materials rather than a low resistant one.

Elements are released from metal restorations under physiological conditions, but their release are augmented under bleaching conditions [14, 17]. The effect of 10% CP and 10% HP on Hg, Ag, Sn, and Cu, which were released from dental amalgam, was studied by electrochemical method (elemental release study) and SEM [28]. The study showed that the long treatment by bleaching agents causes change in the crystalline structure of amalgam as a result of released ions from corrosion process. Because elemental release has been associated with biological risks, bleaching appeared to increase the biologic liability of nickel-based alloys [29].

Products are currently available for the mouthguard bleaching technique, 10% and higher concentrations of HP and CP with a pH close to neutral. CP (10%) is unstable and disassociates into 3% HP and 7% urea [30]. It means that both HP and CP bleaching agents have the same active agent. The extracting solutions in this study were based on home-bleaching procedures using 10% HP or CP. We used HP and CP in solution rather than in a gel form, which is how this agent is normally delivered for mouthguard bleaching. This was done with the purpose of eliminating most variations that may exist between commercial products due to differences in composition, thickness, pH, and decomposition by-products [14]. PBS was used for convenience and to help correlate with previous studies [23]. The results of the current study indicated that increasing peroxide concentration led to an increase in mass released (α=0.05) compared to control group (Table 2). In accordance with the proportion of active agents between the HP and CP solutions, mass released for the HP-treated group was approximately three times as much as compared to the CP-treated one. Comparing the effects of the two bleaching agents at 10% showed that the alloy suffered less corrosion rates in the presence of CP than HP. These results agreed with the SEM observations. However, differences in ion release pictures obtained from SEM analysis were slightly prominent, even though ion release profiles suggested differences between bleaching systems This behavior could be attributed to the high electron density on nitrogen and oxygen active centers of CP that absorbed on the metallic surface, forming a protective layer for corrosion [19].

Although the stability of Ni-based alloys has been shown to be significantly reduced when placed in more acidic environments and metal ion release is thus enhanced [3], the pH was 6.5 in the current study because popular

Table 4 XPS depth profile analysis of the surface oxide layer before and after elemental release measurements, showing the composition content in atomic percent (at.%)

	Ni_{2p}	Cr_{2p}	Mo_{3d}	Si_{2p}	O_{1S}	Nb_{3d}	Ce_{3d}	Fe_{2p}
Before immersion	33.8	7.3	1.95	–	26.4	–	–	–
HP immersion	2.8	8.05	4.1	5.7	66.8	0.02	8.3	0.2
CP immersion	0.4	1.5	5.7	6.2	62.4	0.02	0.1	0.2

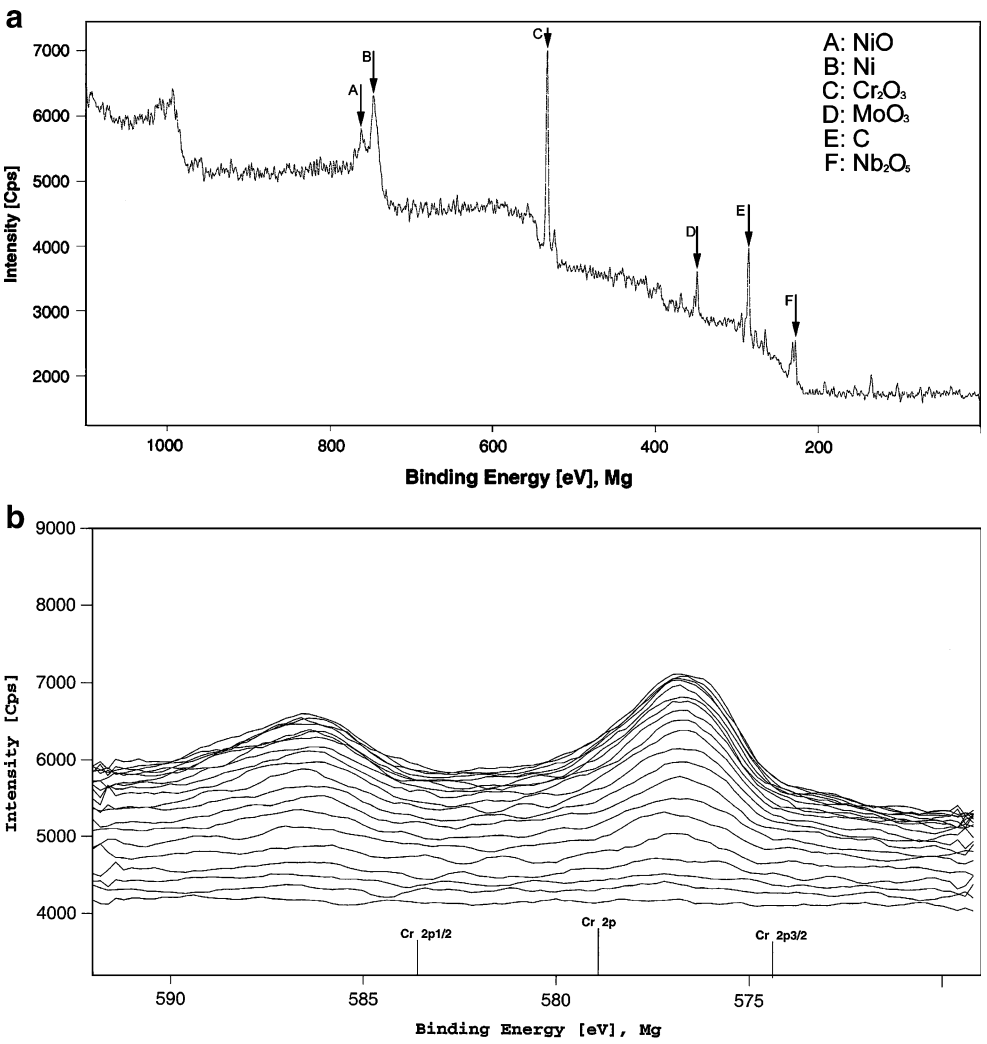

Fig. 2 Representative XPS survey scan. **a** An XPS widescan of CP-treated group. **b** An XPS narrow scan of untreated group showing Cr peak spread over 573.1–576.7 eV and covering Cr^0 and Cr_2O_3 and showing that elemental oxidation can be in various forms

commercial preparations have an average pH of 5 to 6.5 [17]. Since home bleaching comprises daily applications of 5 to 8 h for 2 to 5 weeks [15, 30], in this study, total testing period was selected as 30 days, and the bleaching agents were applied for only 8 h a day in order to simulate the clinical situation as closely as possible.

Intraoral corrosion rates may possibly be predicted from in vitro tests. Gettleman et al. [31] used a linear polarization

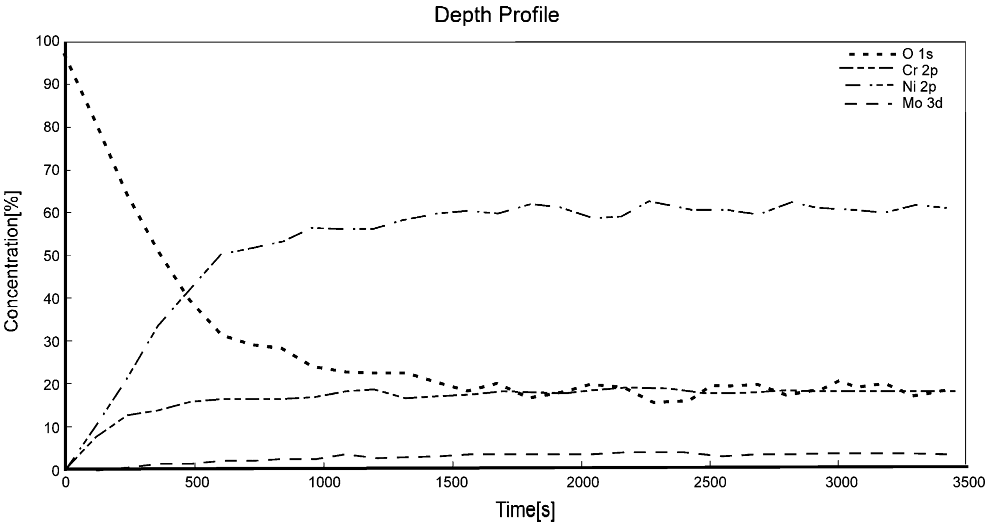

Fig. 3 Representative XPS depth profile analysis of oxide film formed on the surface of control group

technique to study intraoral corrosion phenomena. In their study, measurement of corrosion rates in baboons was correlated with in vitro tests in artificial saliva. Their study included amalgams, casting gold and base metal alloys, stainless steel, and titanium alloys. The in vivo currents measured exceed, in most cases, the corresponding measured in vitro currents. In vivo currents up to about ten times the corresponding in vitro currents were registered in that study. On the contrary to the present study, the results obtained by Johansson et al. [22] showed that Ni–Cr alloy in 0.9% NaCl solution and artificial saliva solution was resistant to corrosion, but the amounts of elements released to immersion solution were not measured in that study. Wataha et al. [23] reported that for Ni-based alloys, release of nickel reached 600–800 μg/cm^2 in case of alloys that were brushed in solutions of PBS at pH 7 with toothpaste. It has been shown that the amount of mass loss was 0.54–3.261 μg/cm^2 from Ni–Cr alloys which were examined using a solution of 0.1 mol/L lactic acid and 0.1 mol/L sodium chloride after 35 days [21]. In the current study, both control solution and test solutions caused elemental release from the tested alloy; however, elemental release increased as a result of bleaching application compared to control group (Table 2). It is difficult to make direct comparisons between the data from the current study and published ones, due to limitations of either differences in test conditions like extracting solutions and the composition of the alloys used or presentation of data in these papers.

The release of metal ions, especially nickel from dental alloys, is a possible concern, because of its local and systemic toxic, immunogenic, mutagenic, and chemotactic effects. The potential biologic hazards, hypersensitivity in particular, from nickel containing dental alloys are still uncertain [32]. Nickel might activate monoyctes and endothelial cells, depending on the ionic concentration [33]. Additionally, nickel complexes in the form of arsenides and sulphides are known to be carcinogenic, allergenic, and mutating substances, whereas nickel at nontoxic concentrations was found to induce deoxyribonucleic acid (DNA) alterations, mainly by base damage and site-specific DNA strand scission [34].

XPS data showed that oxide layers formed on all groups contained mainly Cr_2O_3, NiO, and MoO_3 (Table 3). Since the O concentrations were approximately same, the amount of oxides formed on both HP- and CP-treated samples were almost same, but the composition of the oxides were different (Tables 3 and 4). For HP-treated group, additional oxide forms (Ni_2O_3, Mo_2O_5, CrO_3, and NbO_2) were seen. Moreover, Cr and Ni levels were higher than CP-treated sample. In addition, significant amount of Ce (8.3 at.%), which can easily connect to O, was also demonstrated. XPS data confirmed the oxidizing effect of HP. Except for trace elements, only the Mo level was increased with CP compared to HP treatments. These conflicting results between the XPS and atomic absorption spectrometry (AAS) analyses may indicate the instability of passive film that formed after high concentrated HP (10%) treatment. HP may have accelerated the degradation of the alloy surface by affecting protective surface film; thereby, inner layers of alloys could be damaged. And the results also showed that the presence of especially Mo on the oxide layer as well as Cr improved the growth of oxide layer, leading to a better corrosion behavior.

From our XPS results, it appears that both CP and HP can cause changes in surface levels of the elements. Such changes may be caused by enhanced oxidation, corrosion, and dissolution of the alloy surfaces that take place during exposure to HP and other decomposition by-products of the bleaching agents.

Mouthguard bleaching is usually being performed for 8 h a day and when the patients are asleep where the flow of saliva decreases. One should consider that if the patients have full-cast and/or porcelain fused to metal (PFM) restorations, bleaching treatment must be applied very carefully because excessive material may contact with these restorations and alter the corrosion properties and/or tissue responses, and CP should be selected for bleaching applications rather than HP bleaching agent.

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Conflict of interest The authors declare that they have no conflict of interest.

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