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Elemental Release from CoCr and NiCr Alloys Containing Palladium

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Abstract

Purpose: An entirely new subclass of casting alloy composition whereby palladium (∼approximately 25 wt%) is added to traditional base metal alloys such as CoCr and NiCr was recently introduced to the market. The purpose of this study was to evaluate the elemental release of new CoPdCr and NiPdCr alloys and compare them to traditional CoCr and NiCr alloys.
Materials and Methods: Five casting alloys were investigated: CoPdCr-A (NobleCrown NF, The Argen Corporation), CoPdCr-I (Callisto CP+, Ivoclar Vivadent), NiPdCr (NobleCrown, Argen), CoCr (Argeloy N.P. Special, Argen), and NiCr (Argeloy N.P. Star, Argen). Rectangular specimens (n = 6/alloy) were prepared and immersed in a lactic acid/NaCl solution at 37°C for 7 days according to ISO 10271. Solutions were analyzed with ICP-AES to determine elemental release. The concentrations of major ions (cobalt, nickel, palladium, chromium, and molybdenum) were compared using a generalized linear model ($p < 0.05$). Representative specimens were examined with optical microscopy before and after immersion.
Results: The CoPdCr alloys released a significantly greater amount of respective ions (Co, Cr, Mo, and total ions) compared to the traditional CoCr alloy. No significant differences in elemental release were noted between NiPdCr and NiCr. Optical microscopic examination showed abundant areas of corrosion in the palladium-containing CoCr alloys after immersion, whereas little difference was observed for the other alloys.
Conclusions: Corrosion resistance measured via elemental release was compromised when CoCr was alloyed with palladium, but this effect was not observed with NiCr.

The American Dental Association (ADA) classifies dental alloys for fixed prosthodontics into four groups based on composition. These alloy groups are labeled as high noble, titanium and titanium alloys, noble, and predominately base.[1] The noble alloys must contain at least 25 wt% noble metal (gold, palladium, platinum, rhodium, iridium, osmium, and ruthenium), although these alloys generally contain a significantly greater percentage of noble metal than required.[2] Predominantly, base metal alloys, by definition, contain less than 25 wt% noble metal, but in practice contain little, if any, noble metal content.[2] Recently, a new subclass of noble metal casting alloys has been introduced to the dental market (Callisto CP+; Ivoclar Vivadent Inc., Amherst, NY). This alloy is based upon cobalt-chromium but with 25 wt% Pd. Shortly, another manufacturer released a nickel–chromium based, Pd-containing alloy and its own CoCr-based, Pd-containing alloy (Nobel Crown and Nobel Crown NF, The Argen Corp., San Diego, CA). These new noble alloys are unique in that they evolved from an existing base metal class. Their appeal stems from their relatively lower cost compared to other dental noble alloys despite a relative lack of material property information available on these new alloys.

When selecting a dental alloy, various material properties must be considered to provide the patient with the best restoration for the given situation. For example, grain size, phase structure, yield strength, hardness, elastic modulus, color, porcelain-bonding, and corrosion are among the properties and characteristics that must be balanced.[2] However, it has been suggested that corrosion is the single most relevant property to the biological safety and success of the prosthesis.[2,3] Metallic corrosion is an oxidation process in which an electrochemical reaction causes a dissolution of the metal, and it may exist in many different forms including general corrosion, galvanic corrosion, crevice corrosion, pitting, selective dissolution, and intergranular corrosion.[4] Despite the mode of the process, the significance lies in that some elements of the alloy are released and are therefore in contact with the local hard and soft tissues, as well as the gastrointestinal tract, where they may cause toxic, inflammatory, allergic, or mutagenic reactions.[3]

The biologic response of the surrounding tissues to the alloy is dependent upon the element released, the amount of its release, and the duration of exposure.[3] However, elemental release may not be proportional to the elemental composition of the alloy. A standardized protocol described in ADA Specification No. 5 for Dental Casting Alloys and ISO 10271:2001 "Dental metallic materials-Corrosion test methods"[5,6] involves a static immersion test in which the alloy is immersed in a lactic acid/salt solution to simulate conditions related to plaque accumulation areas with low pH. Thus, the concentrations of metal ions in the solution after immersion may reflect the probable release of ions in the mouth. This method has been used in previous studies to identify and quantify elements released from various dental alloy composition classes.[7–10] However, the elemental release of the new Pd-containing CoCr and NiCr alloys has not yet been determined. This information is needed, as the biocompatibility of some of these alloys' constituents has been questioned in the past.[11–13]

It is generally assumed that alloys with lower noble metal content have inferior corrosion resistance. In an extensive study, Manaranche and Hornberger found that Pd-base and AuPtPd dental alloys were the most resistant, and base metal alloys typically the least resistant to electrochemical corrosion.[9] Thus, the addition of Pd to CoCr and NiCr alloys could be expected to increase their corrosion resistance. Recent in vitro evidence, however, has shown the opposite to occur. Sarantopoulos et al found that CoPdCr and NiPdCr alloys had decreased polarization resistance and increased corrosion current densities compared to their CoCr and NiCr counterparts.[14] A limitation of electrochemical testing, although, is that the quantity and identity of the released ions is not known. Therefore, the aim of this study was to further investigate the corrosion properties of these three new casting alloys by comparing the elemental release of CoPdCr and NiPdCr alloys to that of existing CoCr and NiCr alloys. The null hypothesis was that the inclusion of Pd into CoCr and NiCr alloys has no effect on elemental release.

Materials and methods

Three noble alloys consisting of two palladium-containing cobalt-chromium (CoPdCr) and one palladium-containing nickel–chromium (NiPdCr) alloy were evaluated and compared to traditional NiCr and CoCr base metal alloys. The compositions (in wt%) of these alloys are listed in Table 1. The elemental release of the five dental casting alloys was determined following the static immersion test method in ISO 10271:2001 "Dental metallic materials-Corrosion test Methods."[6] Six specimens per alloy composition were cast in accordance with the manufacturers' recommendations into $27 \times 12 \times 3$ mm^3 rectangles using sectioned red boxing wax as a casting pattern. The wax rectangle sections were sprued, invested in a carbon-free, phosphate-bonded investment (Formula 1; Whip Mix Corp, Louisville, KY), and burned out. Casting was performed by a private dental laboratory (Capitol Dental Lab, Menomonee Falls, WI) using individual quartz crucibles for each alloy, a multiorifice gas-oxygen torch, and centrifugal casting machine. After divestment, the sprues were removed, and the specimens were blasted with 125 μm alumina. The specimens were then degassed/oxidized according to the manufacturer's instructions using a computerized porcelain furnace (Centurion VPC; Ney, Yucaipa, CA). For CoPdCr-A, CoCr, NiPdCr, and NiCr, the specimens were fired from 650°C to 980°C at 55°C/min under vacuum. The CoPdCr-I specimens were fired from 650°C to 900°C at 55°C/min and held for 1 minute without a vacuum. Next, as specified in ISO 10271 to simulate the heat treatment associated with a porcelain firing schedule, all specimens were fired from 600°C to 930°C at 55°C/min and held for 10 minutes in air.

All specimens were ground following standard metallographic procedures with FEPA 1200-grit silicon carbide paper used for final grinding. The surface area of each specimen was determined using measurements with a vernier caliper. Specimens were sonicated (Ultrasonic T-15; L&R Manufacturing Co, Kearny, NJ) for 2 minutes in methanol, rinsed with water, and dried with oil-free, compressed air. An immersion solution was prepared by dissolving 10.0 g 90% $C_3H_6O_3$ (lactic acid) and 5.85 g NaCl in 1000 mL of water. The pH of the solution was tested (inoLab Level 3; WTW GmbH & Co., Weilheim, Germany) and found to be 2.3. Each specimen was placed in a polypropylene centrifuge tube (Corning Inc., Corning, NY) and filled with enough of the lactic acid/sodium chloride solution to achieve a ratio of solution to surface area of 1 ml:1 cm^2. Three solutions without a specimen (blank) were also prepared. The specimens in the solution were stored upright in an incubator at 37°C for 7 days and then removed from the test tubes. The residual solution was analyzed with inductively coupled plasma-atomic emission spectroscopy (ICP-AES) for the elemental release of Co, Ni, Cr, Pd, and Mo measured in μg/g. The detection limit of each element in solution was <0.05 μg/g. Elemental solution concentration was converted to elemental release per surface area over immersion time, or mg/(cm$^2 \cdot 7$ days).

Before and after immersion testing, representative specimens from each group were examined with a metallurgical microscope (Olympus PME3; LECO Corp., St. Joseph, MI) with images captured via a digital image acquisition device (SPOT Insight 2MP Firewire Mono; Diagnostic Instruments Inc., Sterling Heights, MI) and software (SPOT Software 4.5; Diagnostic Instruments Inc.). Elemental release data for each alloy was compared using a generalized linear model in SAS (SAS Institute Inc., Cary, NC) to look at fold changes between alloy pairs for each element. The Benjamini-Hochberg method, which is designed to control the false discovery rate, was used to control type I errors associated with multiple testing with significance set to $p < 0.05$.

Table 1 Elemental compositions (wt%) of the alloys provided by the manufacturers

			Composition (wt%)					
Alloy name	Code	Manufacturer	Co	Ni	Pd	Cr	Mo	Other
NobleCrown NF	CoPdCr-A	The Argen Corporation (San Diego, CA)	45	–	25	20	10	B
Callisto CP+	CoPdCr-I	Ivoclar Vivadent, Inc. (Amherst, NY)	40	–	25	21.4	12.7	<1 W, B, Ta
Argeloy N.P. Special	CoCr	Argen	59.5	–	–	31.5	5	2.0 Si, 1.0 Mn, 1.0 Other
Argeloy N.P. Star	NiCr	Argen	–	61.2	–	25.8	11	Al, Mn, 1.5 Si
Noble Crown	NiPdCr	Argen	–	37.5	25	25	12	Si

Results

Table 2 lists the elemental release in mg/(cm^2 · 7 days) for each alloy group. The CoPdCr alloys released a significantly ($p < 0.05$) greater amount of Co, Pd, Cr, and Mo compared to the traditional CoCr alloy. No significant differences ($p > 0.05$) were noted between NiPdCr and NiCr. Not surprisingly, the total ion release, given as the sum of the values for each of the five elements, was also significantly ($p < 0.05$) greater for the CoPdCr alloys as compared to all other alloy groups. There was no significant difference in the total ion release of the five ions between CoPdCr-A and CoPdCr-I, as well as between CoCr, NiCr, and NiPdCr. The detection limit for each element corresponded to <0.05 mg/(cm^2 · 7 days), and the ions of interest in the control solutions were all below the detection limit, indicating all ions released originated from the alloy and not extraneous sources.

Figure 1 displays optical micrographs of representative specimens of each alloy before and after immersion in the lactic acid/salt solution for 7 days. CoCr, NiCr, and NiPdCr displayed little change in appearance with immersion, whereas the CoPdCr alloys showed areas of corrosive attack evident as the dark areas on the micrographs.

Discussion

The CoPdCr alloys released significantly greater amounts of the considered ions compared to CoCr, whereas the NiPdCr did not compared to NiCr; thus, the null hypothesis that inclusion of palladium into the respective base metal alloys has no effect on elemental release has been rejected for CoPdCr and accepted for NiPdCr. Similarly following ISO 10271, Manaranche and Hornberger[9] measured the ion release of various alloys and pure metal specimens to develop a chemical classification system for dental alloys. They distinguished three ascending classes (I to III) according to the quantity of metallic ions released and proposed that only class III alloys present a risk of inducing adverse biological reactions in patients. Both CoPdCr alloys in the present study would be considered class II, as they fall within the 10 to 100 mg/(cm^2 · 7 days) range. Interestingly, the CoCr and remaining Ni-based alloys would be considered class I alloys [<10 mg/(cm^2 · 7 days)]. Further, a comparison of the released amount of corrosion products with ISO standard 22674:2006 "Dentistry-Metallic materials for fixed and removable restorations and appliances"[15] for maximum ion release after 7 days showed that the limit of 200 μg/cm^2 was not reached by any of the alloys tested. These considerations suggest that whereas there was an increase in elemental release for the CoPdCr alloys, the clinical significance of the amount of ions released may be questioned. On the other hand, pinpointing a specific threshold amount of released elements sufficient to cause adverse biological events remains elusive.

Following reports expressing concern with Pd-containing alloys with regard to allergy and adverse biological effects,[16,17] the biocompatibility of Pd-containing dental alloys became a topic of great interest beginning in the 1990s. The frequency of Pd sensitivity ranges from 2% to 18%,[17] and it is believed that the Pd^{2+} ion, rather than pure Pd, causes sensitization and allergic reactions.[18] The extent of adverse biological effects from Pd-containing alloys remains controversial,[17,18] but it seems the scope of the problem may be alloy specific,[19] and the amount of Pd release is dependent upon other alloying elements. For example, Tufekci et al[7] measured the in vitro elemental release from PdCuGa and PdGa alloys at 7, 70, and 700 hours and

Table 2 Elemental release in mg/(cm^2 · 7 days) [mean (standard deviation)]

Alloy	[Co]	[Ni]	[Pd]	[Cr]	[Mo]	[Total]
CoPdCr-A	50.4 (24.9) A	BDL	0.4 (0.4) A	11.6(8.2)A	26.8(3.6)A	89.1(35.8)A
CoPdCr-I	33.3 (9.8) A	BDL	0.3 (0.5) A	7.2(4.0)A	29.4(9.0)A	70.2(18.9)A
CoCr	2.9 (2.5) B	BDL	BDL	0.3(0.2)B	1.6(0.8)C	4.8(3.4)B
NiPdCr	BDL	2.3 (0.4) A	BDL	0.4(0.1)B	3.6(0.7)B	6.3(1.2)B
NiCr	BDL	3.2 (2.2) A	BDL	0.5(0.3)B	1.3(0.7)BC	5.0(3.0)B

BDL = below detection limit [<0.05 μg/(cm^2·7 days)]. [Total] represents the sum concentration of the 5 individual ions. The concentrations of ions in the control solutions (without an alloy specimen) were all below the detection limit. Different letters denote significant differences ($p < 0.05$) between alloys.

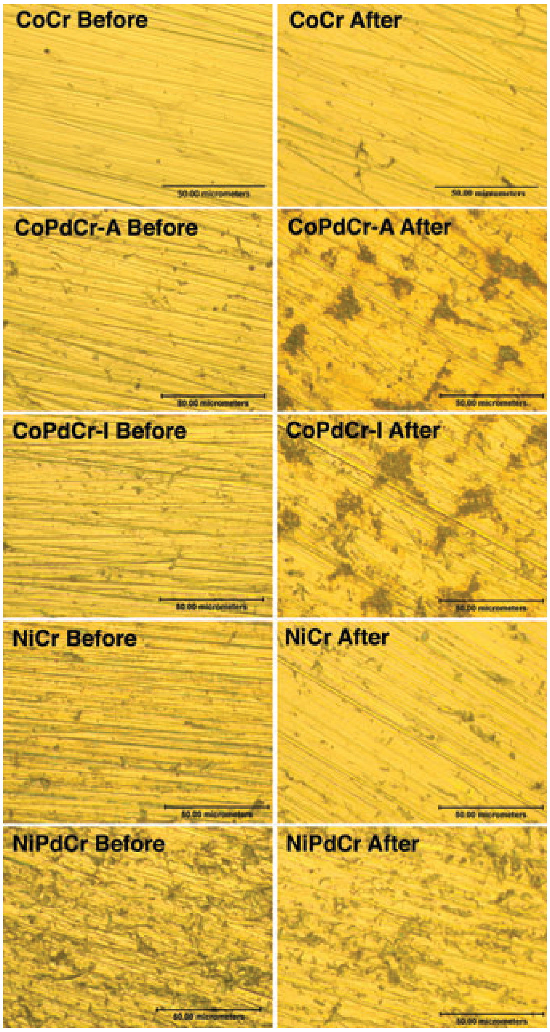

Figure 1 Micrographs of alloys before (left column) and after (right column) immersion testing. CoCr, NiCr, and NiPdCr displayed little change in appearance with immersion, whereas the CoPdCr alloys showed areas of corrosive attack evident as the dark areas on the micrographs.

found the mean concentration of released palladium increased with time and was approximately 97 μg/cm^2 and 5 μg/cm^2, respectively, at 700 hours. The increased ion release with time was consistent with results from Wataha and Lockwood,[20] who had earlier reported that the release of elements from dental alloys continues through 10 months, but at a constant rate after around 2 months. Syverud et al[10] compared the elemental release of a PdCuGa alloy with a PdGa alloy prepared with different thermal oxidation and/or grinding surface conditions. They found that the copper-containing alloy leached much greater amounts of Pd and total ions, and the dissolution rate depended upon oxidation history and oxide layer thickness. These studies indicate that the overall elemental composition, various phases formed, thickness of the oxide layer, and duration of immersion all contribute to the amount of ions released into the testing medium. The amount of Pd released from the CoPdCr alloys in this study seems to be comparatively low,[7,10] although one must acknowledge the different percentage of Pd contained in the alloys is a factor in this observation. Selective dissolution seems to have occurred for Co and Mo, but not Pd and Cr, based upon the relative amount of ions released [individual ion concentration/total ion concentration released; e.g., (Co/Total) compared to the alloy composition (Table 1)]. This suggests different phases may exist in the microstructure of the alloy with the Pd- and chromium-rich areas acting cathodic to Co- and Mo-rich phase areas. A similar type of phenomenon has been observed in PdCuGa alloys, where a eutectic Pd_2Ga phase was anodic to a more noble Pd solid solution phase,[18] which would explain the differing ion release between PdCuGa and PdCu alloys mentioned above. Alternatively, the more active constituents (e.g., Co and Mo) in a Pd-rich phase may selectively leach, resulting in increased corrosion of that element but lesser Pd release. Less focus on the elements released in greater quantity (Co and Mo) appears in the literature, although 8% of the general population is sensitive to Co.[17]

The increase in elemental release with the CoPdCr alloys in this study may be related to the affect Pd has on the microstructure of the alloys. The microstructure of CoCr (and NiCr) alloys is dendritic and becomes more complex with the addition of alloying elements by formation of precipitates and/or secondary phases.[21] If the composition of the precipitates and phases has a decreased Cr content, its protective passive layer may be unstable or not present. Espevik[22] showed that the release of Ni and Cr from base metal alloys was not observed when greater than 27% Cr was present; however, when the Cr content of the alloy was below 16%, significant amounts of Ni and Cr were released. Wylie et al[23] also concluded that the presence of higher Cr (25 wt%) content in Ni-based alloys led to superior corrosion resistance compared with lower Cr (12.6 wt%) content. A comparison is difficult, considering the differing alloy systems, but it is generally regarded that at least 12 wt% Cr is needed to establish passivity.[4] Thus, it is hypothesized that by adding Pd into the CoCr alloy system, a phase, or phases, deficient in Cr may arise in the microstructure and decrease corrosion resistance overall. A similar occurrence of local depletion of Cr has been observed with NiCrBe alloys, where an NiBe phase lacked sufficient amounts of Cr to establish passivity for that phase.[24,25] Compounding this is that Pd and/or Pd-rich areas would naturally be cathodic to these as mentioned above. The microscopic areas of corrosive attack noted in the CoPdCr alloys (Fig 1) supports the hypothesis that regions of different phase composition are acting anodic relative to others. Further research into the microstructure and phase composition in the CoPdCr alloys is currently underway to investigate this hypothesis.

The results of this study showed that there was no significant difference in the elemental release of the NiPdCr alloy compared to the NiCr alloy. It is possible that the use of a greater sample size may have revealed significant differences between NiPdCr and NiCr, but still the difference in mean elemental release was below 2.5 μg/(cm^2 7 days) for all ions, individually or combined. It is unlikely this small amount would be clinically significant. These results for NiPdCr versus NiCr were surprising, considering that electrochemical testing reported by Sarantopoulos et al[14] revealed decreased in vitro corrosion resistance for the same Pd-containing alloys in a phosphate-buffered saline solution (PBS). Specifically, they observed a decrease in polarization resistance, an increase in current density, and a breakdown of the passive layer resulting in pitting of the Pd-containing CoCr and NiCr alloys after potentiodynamic testing; however, it has been shown that the corrosion testing environment may influence corrosion behavior and the production of corrosion elements. For example, Denizoğlu et al[26] compared ion release of an NiCr alloy in artificial saliva solutions of pH 4, 5, and 7 and found Ni ion and total ion release was lower in the pH 5 solution than in the pH 7 solution. Conversely, Covington et al[27] and Wataha et al[28] demonstrated increased Ni release from NiCr with decreasing pH levels in their testing solutions. The discrepancy in results between the two studies for the NiPdCr alloy may arise because of the difference in testing medium pH and composition affecting the alloy in dissimilar ways.

As previously mentioned, the release of ions is a necessary, although not solely sufficient, event for allergic, toxic, and mutagenic reactions to occur, and it has been suggested that clinicians should select alloys that have the lowest release of elements to minimize biological risks.[3] Thus, the increased release of ions from the CoPdCr alloy as compared to the CoCr alloys could raise concern for the biocompatibility of this new subclass of casting alloy composition. Although the frequency of adverse reactions with prosthodontic materials is relatively low (0.25% of patients), reactions may be local, such as lichenoid reactions, or more general, as observed by skin eruptions.[12] Specifically, allergic reactions would present as redness, swelling, or mucosal erosion.[12] However, the correlation of ion release to biological consequence is difficult with such a static immersion test for various reasons. First, this study followed ISO 10271 and used a lactic acid/NaCl test medium. How this testing medium compares to the actual intraoral conditions is not a straightforward matter, and the ideal in vitro test medium has not yet been determined. Similarly, as mentioned previously, surface condition of the alloys will affect elemental release.[10] The alloys were ground according to ISO 10271 to simulate finishing and polishing the alloys, but in a clinical situation, some areas of the intaglio surface of a crown, for instance, may or may not be airborne particle abraded, resulting in altered amounts of oxide removal and ultimately ion release. Further, this study is a short-term test (1 week), and Wataha et al[29] demonstrated that long-term elemental release evaluations may be more appropriate because of changing rates of elemental release over time. They also noted that this changing rate of ion release further complicates the relationship between elemental release and cytotoxicity. Of most importance, the dynamic nature of the intraoral conditions makes in vitro studies insufficient in predicting clinical results. Dental alloys are subjected to a varying chemical environment and also experience mechanical disruption in the form of occlusal and toothbrushing forces. Thus, clinical evaluation is required before the safety of Pd-containing CoCr and NiCr alloys can be fully determined.

Conclusion

Corrosion resistance measured via elemental release was compromised when CoCr was alloyed with palladium, but this effect was not observed with NiCr.

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