Effect of pH on element release from dental casting alloys

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Statement of problem. Dental casting alloys are subjected to transient acidity in the oral environment, yet most studies have not investigated the effects of these transient environments on elemental release from alloys. Elemental release is important because it plays a significant role in alloy biocompatibility.

Purpose. It was hypothesized that acidic environments would increase elemental release from dental alloys during exposure and after the acidic environment was removed. This hypothesis was based on the known increase in release of nickel from nickel-based alloys in an acidic environment.

Material and methods. High-noble, noble, and base metal casting alloys were exposed for 30 minutes to solutions with pH ranging from 1 to 7. Elemental release of representative elements was measured by means of atomic absorption spectrometry during the exposure and in the week after the exposure. This release was compared with elemental release in the week before the exposure.

Results. High-noble and noble alloys were resistant to acidic environments. A pH of 4 did not increase elemental release during or after exposure. A pH 1 environment slightly elevated release of Ag, Cu, and Pd in some alloys. However, a Ni-based alloy released large amounts of Ni during the acidic exposure of pH 1 or 4, and more importantly, in the week after the exposure as well. Increased time of exposure to acid did not alter elemental release from noble or high-noble alloys, but markedly increased release from the Ni-based alloy.

Conclusions. Transient exposure of casting alloys to an acidic oral environment is likely to significantly increase elemental release from Ni-based alloys, but not from high-noble or noble alloys. (J Prosthet Dent 1998;80:691-8.)

CLINICAL IMPLICATIONS

The current study has shown that exposure of Ni-based alloys to transient periods of biologically relevant levels of reduced pH can significantly increase Ni release from the tested alloy. High-noble and noble alloys were resistant to this effect and probably release a lower amount of elements into the body locally and systemically compared with Ni-based alloys. This decreased burden on the body may be important in any biologic effect that the alloys have.

Cytotoxicity tests are used to estimate the biologic safety of dental casting alloys. Studies have reported the cytotoxicity of high-noble alloys [1-3] and noble[4] and predominately base metal alloys.[5,6] The biologic safety of combinations of alloys,[7] and newer tests such as coculture of 2 types of cells[8] and the use of human cells[9] to measure alloy cytotoxicity have also been reported. Other research has focused on the relationship between cytotoxicity and release of elements from alloys. The current consensus is that in vitro, element release from alloys correlates with cytotoxicity, but the relationships between release of elements and cytotoxicity are often complex.[10-14]

The release of elements from dental casting alloys has been more extensively investigated than cytotoxicologic effects. The motivation for studying elemental release is primarily its relationship to alloy biocompatibility. Elemental release has been reported for high-noble and noble alloys,[15-17] base metal alloys,[17,18] and for other types of alloys and solders.[19-21] Most of these tests have focused on measurement of release during the exposure to a biologic medium or artificial saliva over periods ranging from 24 hours to 1 month. One study reported the effect of periodically changing the medium on element release over a 10-month period.[22]

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Table I. Compositions of alloys (weight percent)

Alloy	Ag	Au	Cu	Cr	Ga	In	Mo	Ni	Pd	Zn	Other
High-noble (Au-Pt)	9.2	73.8	4.4			15				2.0	Ir 0.1 Pt 9.0
High-noble (Au-Pd)		51.1			1.2	9.0			38.5		Ir 0.2
Noble (Pd-Cu-Ga)		1.0	5.0		6.0				79.7		Pt 1.0, Ru 0.8, Sn 6.5
Base metal (Ni-based)				15.0			5.0	76.0			Be 1.8 B tr

Table II. Detection limits for elements using ICP-MS (ng/cm^2) or AAS (μg/cm^2)*

Alloy	Ag	Au	Cu	Cr	Ga	In	Mo	Ni	Pd	Zn
ICP-MS	0.012	0.005	0.020	0.060	0.015	0.006	0.005	0.033	0.023	0.056
AAS	0.008		0.015					0.005	0.056	0.002

**ICP-MS*: Inductively coupled mass spectroscopy. *AAS*: Flame atomic absorption spectroscopy, adjusted for surface areas and volumes used in the current study to make units appropriate to figures.

In most of these studies, initial element release (first 24 hours) is relatively high, followed by a gradual equilibration to a more constant rate over several weeks.[21,22]

The effect of a constant reduced pH on elemental release from Ni-based alloys has been reported to increase Ni release.[18,23] However, the effect of reduced pH on high-noble and noble alloys, especially newer formulations developed in the past decade, is not well-documented, and the effect of more transient changes in pH on element release is not known. In the mouth, alloys may be exposed to transient pH changes either from foods or plaque.[24]

The purpose of this study was to determine whether a short-term (30 minutes) exposure of high-noble, noble, and base metal casting alloys to a reduced pH increased the release of elements during the exposure and after a return to neutral pH. On the basis of the increased elemental release from Ni-based alloys during exposure to lower pH, it was hypothesized that reduced pH would increase element release from other types of alloys as well. Furthermore, because the reduced pH probably acts by altering the alloy surface,[19,25] it was hypothesized that release of elements subsequent to the low pH exposure would also be higher as the surface reequilibrated in the neutral environment. Therefore this study sought to estimate more precisely the elemental burden that the body faces as the result of dynamic changes in the pH around the alloys. If alloys release more mass from brief exposures to lower pH environments, then the long-term burden of elements released into the body would be substantially increased. This increased burden may alter the biologic response of adjacent tissues to the alloys.

MATERIAL AND METHODS

A high-noble Au-Pt alloy, a high-noble Au-Pd alloy, a noble Pd-Cu-Ga alloy, and a Ni-containing base metal alloy, which are commercially available (Degussa, Hanau, Germany), were used because they represented alloys commonly used in clinical practice (Table I). Alloys were cast into plates 35 mm long, 11 mm wide, and 1.5 mm thick (n = 6). To simulate porcelain firing, alloys were heated to 950°C for 10 minutes, then polished to clinically acceptable surfaces with silicon-carbide paper and, finally, Tripoli and Rouge materials (Schein, Port Washington, N.Y.) on rag wheels. The surface area of each specimen was 9.08 cm^2. After polishing, the alloys were scrubbed with Alconox (Alconox Inc., Alconox, N.Y.) soap, rinsed with distilled water, ultrasonically cleaned for 5 minutes in isopropyl alcohol, and then soaked for 20 minutes in alcohol to disinfect them. Finally, the specimens were rinsed twice with sterile distilled water in a laminar flow hood and dried at 60°C for at least 24 hours. This cleaning process ensured that the alloys were clean enough so that microbial growth did not occur in the extraction medium. These procedures have been used extensively in previous experiments.[3]

After cleaning, the alloys were placed into sterile 15 mL polystyrene centrifuge tubes (Costar, Cambridge, Mass.) such that an insignificant portion of the alloy surface touched the tube (Fig. 1). Seven milliliters of cell-culture medium consisting of Dulbecco's Modified Eagle's medium (Gibco BRL, Grand Island, N.Y.), 3% NuSerum (Collaborative Research, Bedford, Mass.), gentamycin (10 μg/mL), penicillin (125 units/mL), and streptomycin (125 μg/mL) (all from Gibco) cultures were placed into each tube. The medium was selected

as a biologically relevant medium that has been used extensively in previous biocompatibility experiments.[3,15,26] The ratio of the surface area of the alloy to the volume of the medium was 1.3 cm^2/mL, which was midrange (0.5 to 6.0 cm^2/mL) of that required by the International Standards Organization for testing of this type.[27] This initial exposure of the alloys to medium equilibrated the alloy with a biologically relevant environment. Control tubes contained medium with no alloy. The alloys were left in the medium for 1 week and were incubated at 37°C in a 95% air, 5% CO_2 atmosphere.

After the initial medium exposure, the alloys were removed from the tubes, rinsed briefly in sterile water, then transferred to 7 mL of the treatment solution for 30 minutes at 37°C. Four types of treatments were used:

1. Phosphate-buffered saline at pH 7 (Sal-7),
2. 0.1 M lactic acid and 0.1 M NaCl at pH 4 (LA-4),
3. phosphate-buffered saline at pH 1 (Sal-1), and
4. 0.1 M lactic acid and 0.1 M NaCl at pH 1 (LA-1).

The Sal-7 treatment was used as a control against which the other treatments were compared. The LA-4 solution was identical to the solution commonly used in standard corrosion testing,[27] and represents the low end of the pH under active plaque.[24] The Sal-1 and LA-1 solutions were used to test, under extreme conditions, the resistance of the alloys to reduced pH. In selected experiments, the exposure time was increased from 30 to 240 minutes. After the treatment solution was used, the alloys were again rinsed in sterile water, then added to clean tubes containing 7 mL of new cell-culture medium for an additional week. Alloys were repolished and cleaned between the different treatment solutions. The experimental procedure is illustrated in Figure 1.

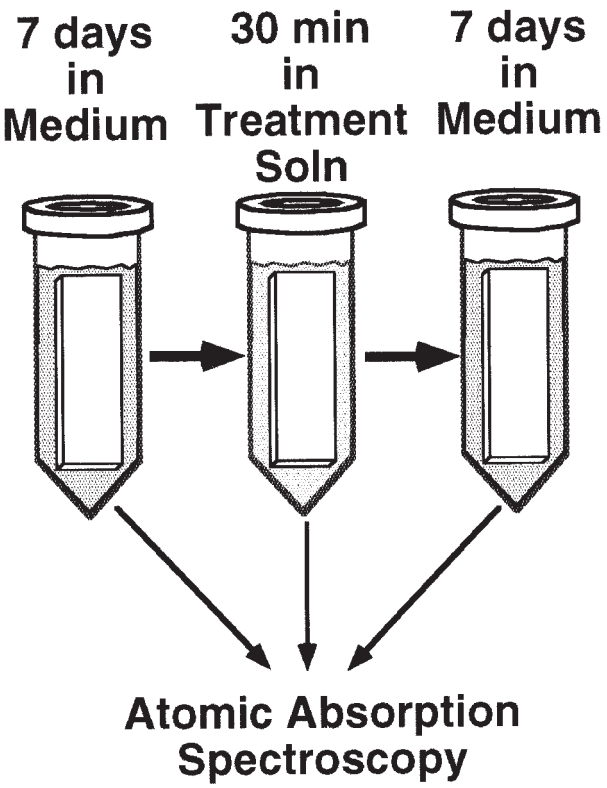

Fig. 1. Diagram of experimental procedure. Alloys were polished and cleaned, then placed into cell-culture medium for 7 days. Next, alloys were exposed to treatment solution of varying pH for 30 minutes. After sterile water rinse, alloys were placed into new cell-culture medium for second 7 days. AAS was used to assess solutions for elements released from alloy.

In pilot experiments, inductively coupled plasma-mass spectroscopy (ICP-MS) was used to assess release of all components of each alloy. This sensitive technique (Table II) was used to determine which elements were primarily released from the alloys. From these pilot experiments, elements with the greatest mass released were chosen to monitor release during the main experiments. Silver, copper, and zinc were chosen for the high-noble Au-Pt alloy, Pd and Cu were chosen for the noble Pd-Cu-Ga alloy, Pd was chosen for the high-noble Au-Pd alloy, and Ni was chosen for the Ni-based alloy. Atomic absorption spectrometry (AAS) was used to determine the release of elements from the alloys into the cell-culture medium or treatment solution. Detailed procedures for this technique have been published previously.[15] Flame AAS was used as opposed to ICP-MS because of the simplicity, speed, and economy of the AAS technique relative to ICP-MS. Detection limits for the AAS technique are presented in Table II.

Results were analyzed by determining the mass loss during the 30-minute treatment of each element per squared centimeter of exposed alloy surface relative to the Sal-7 control group. Mass loss (μg) was estimated by multiplying the concentrations of elements in solution (μg/mL) by the volume of solution (mL). Furthermore, the mass loss from the alloys in the week after the 30-minute treatments was compared with mass loss in the first week of medium exposure. Statistical differences between groups were assessed with 1-way analysis of variance and Tukey multiple comparison intervals (α=.05).

RESULTS

Elemental release during the reduced pH exposure

A reduced pH (Sal-1, LA-1, and LA-4 groups) significantly increased Ag and Cu (P<.05), but not Zn release from the Au-Pt alloy during the 30-minute exposure (Fig. 2). Copper release was only promoted by the lactic acid solution and not by the saline solution at pH 1. At the more physiologically relevant pH of 4, no increase was detected for Ag, Cu, or Zn. For the Pd-Cu-Ga alloy, a pH of 1 significantly (P<.05)

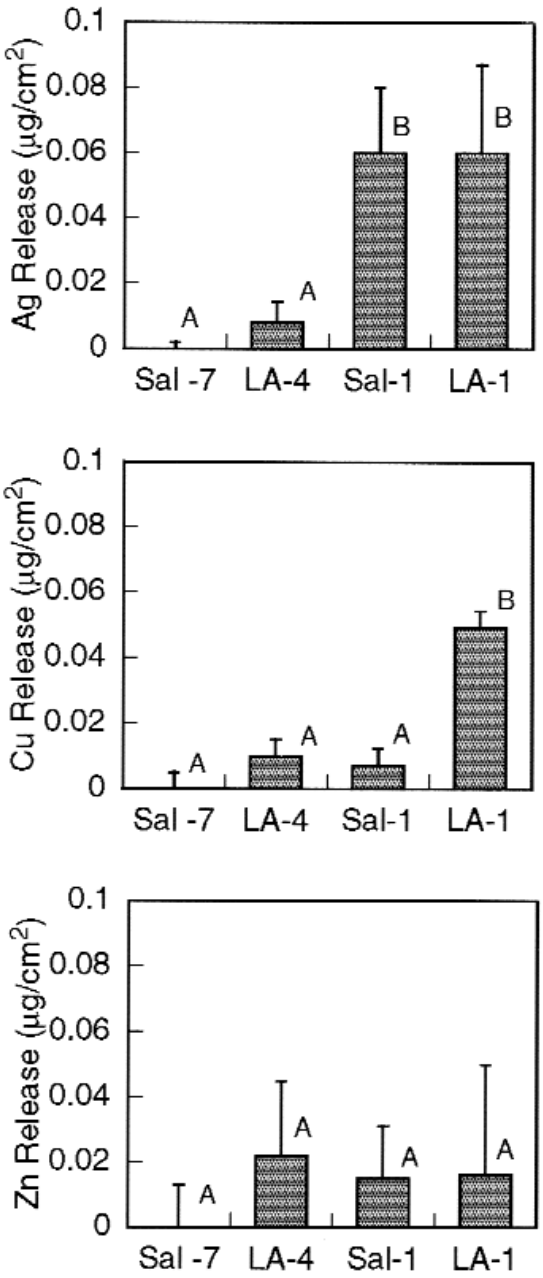

Fig. 2. Elements released from Au-Pt high-noble alloy during 30-minute exposure to (a) saline at pH 7 (Sal-7), (b) 0.1 M lactate at pH 4 (LA-4), (c) saline at pH 1 (Sal-1), or (d) 0.1 M lactate at pH 1 (LA-1). Detection limits for Ag, Cu, and Zn were 0.008, 0.015, and 0.002 μg/cm^2, respectively. Error bars indicate 1 standard deviation of n = 6. Different letters adjacent to columns indicate statistical differences (ANOVA, Tukey, α=.05).

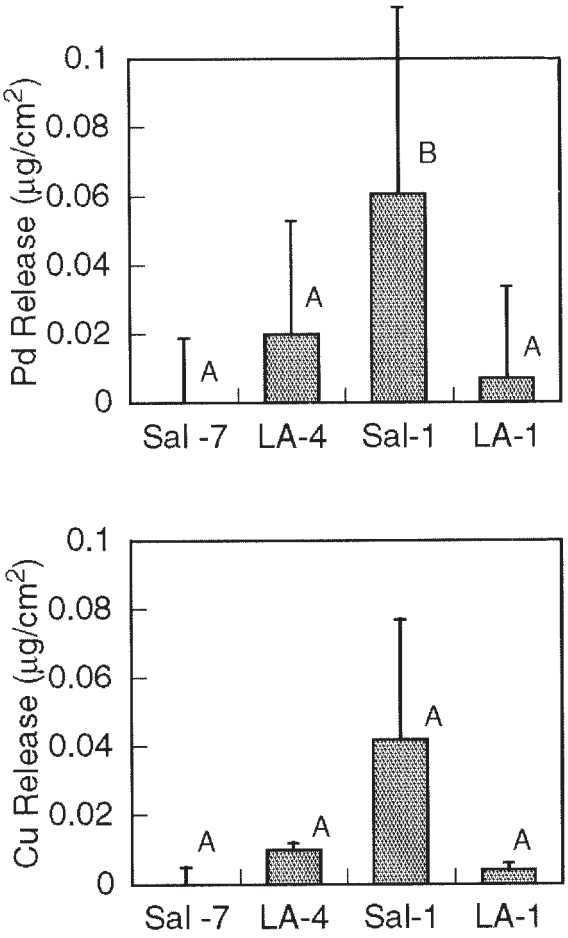

Fig. 3. Elements released from Pd-Cu-Ga noble alloy during 30-minute exposure to (a) saline at pH 7 (Sal-7), (b) 0.1 M lactate at pH 4 (LA-4), (c) saline at pH 1 (Sal-1), or (d) 0.1 M lactate at pH 1 (LA-1). Detection limits for Cu and Pd were 0.009 and 0.056 μg/cm^2, respectively. Error bars indicate 1 standard deviation of n = 6. Different letters adjacent to columns indicate statistical differences (ANOVA, Tukey, α=.05).

increased only Pd release, and the large variation in Pd release made the results less convincing (Fig. 3).

An increase in Cu release was observed with the saline at pH 1, but it was not statistically significant. As with the Au-Pt alloy, a pH of 4 caused no increase in elemental release relative to the pH 7 saline controls. For the Au-Pd alloy, none of the treatment solutions increased Pd release (Fig. 4, top). As expected, reduced pH dramatically increased Ni release from the Ni-based alloy (Fig. 4, bottom). The levels of release during the 30-minute treatments were 1.5 to 3.5 μg/cm^2, which were about 15 times greater than increases seen with the Au-Pt and Pd-Cu-Ga alloy. Conditions at pH 1 caused twice as much Ni to be released as pH 4 conditions. The presence or absence of lactate made no difference, in contrast to the Au-Pt alloy.

Elemental release after exposure to reduced pH

In the week after the exposure to the treatment solution, no increases in element release were observed for

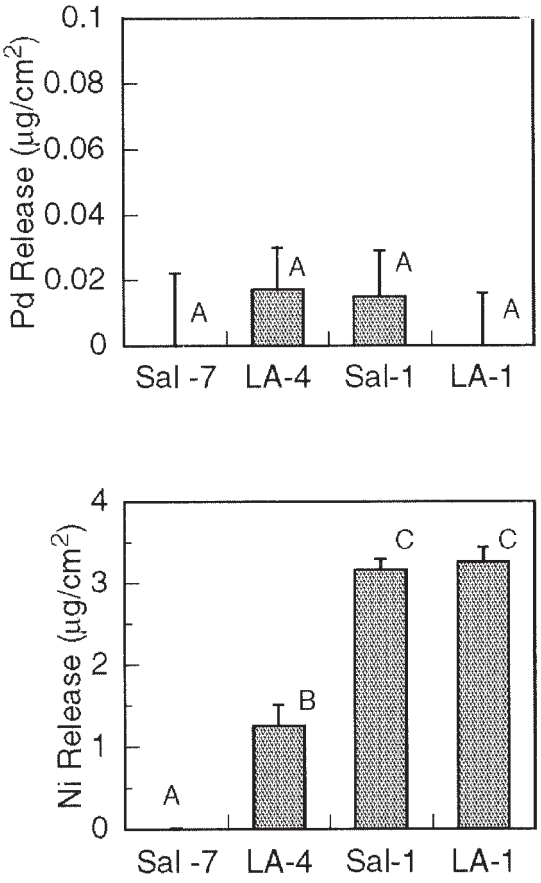

Fig. 4. Elements released from Au-Pd high-noble alloy (top) or base metal alloy (bottom) during 30-minute exposure to (a) saline at pH 7 (Sal-7), (b) 0.1 M lactate at pH 4 (LA-4), (c) saline at pH 1 (Sal-1), or (d) 0.1 M lactate at pH 1 (LA-1). Detection limits for Pd and Ni were 0.029 and 0.005 μg/cm², respectively. Error bars indicate 1 standard deviation of n = 6. Different letters adjacent to columns indicate statistical differences (ANOVA, Tukey, α=.05).

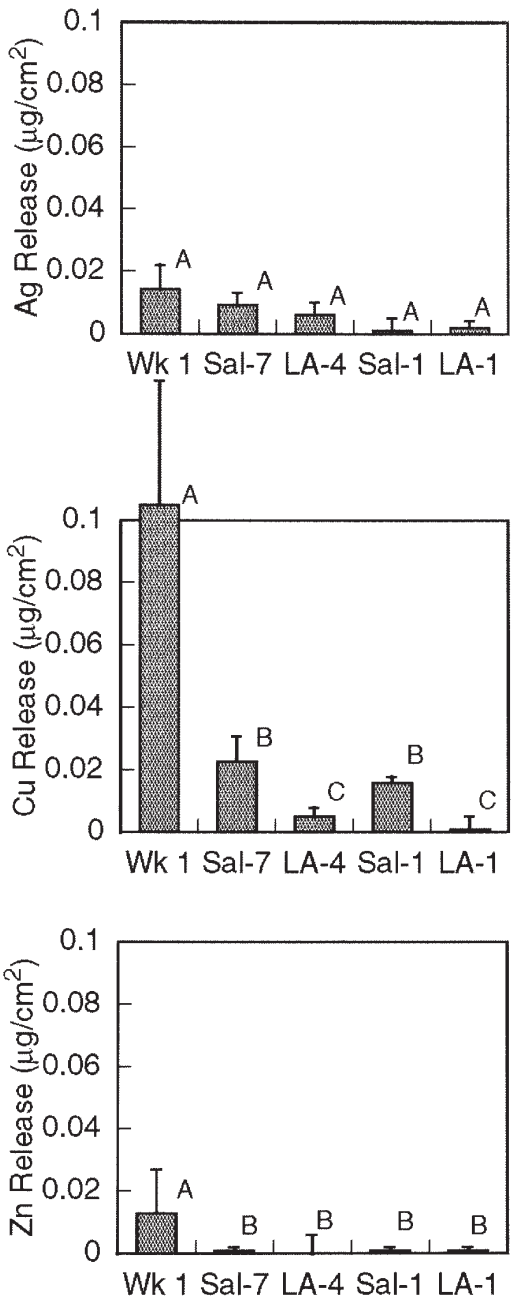

Fig. 5. Elements released from Au-Pt high-noble alloy into cell-culture medium for 1 week before (column labeled Wk 1) and after 30-minute exposure to (a) saline at pH 7 (Sal-7), (b) 0.1 M lactate at pH 4 (LA-4), (c) saline at pH 1 (Sal-1), or (d) 0.1 M lactate at pH 1 (LA-1). Detection limits for Ag, Cu, and Zn were 0.008, 0.015, and 0.002 μg/cm², respectively. Error bars indicate 1 standard deviation of n = 6. Different letters adjacent to columns indicate statistical differences (ANOVA, Tukey, α=.05).

the Au-Pt alloy (Fig. 5). For the pH 7 saline control, release of Cu and Zn were significantly less in the second week of exposure to cell-culture medium than in the first week. Other treatment solutions of reduced pH appeared to reduce Cu release from this alloy in the second week relative to the pH 7 control, and lactate reduced the release significantly at pH 4 or 1 ($P<.05$). For the Pd-Cu-Ga alloy, Pd release was below detection limits for both weeks and all treatment conditions (Fig. 6). Copper release was significantly lower in the second week than the first as expected, and no reduced pH treatment increased or decreased Cu release in the second week. For the Au-Pd alloy, Pd release in the first and second weeks was just above detection limits and was statistically the same (Fig. 7, top). As with Cu in the Au-Pt alloy, a reduced pH significantly reduced Pd release to below detection limits ($P<.05$). For the Ni-based alloy, the release from saline controls was significantly reduced in the second week as expected. However, a 30-minute exposure to pH 4 significantly increased the release of Ni in the second week (Fig. 7, bottom) above the saline controls, and exposure to pH

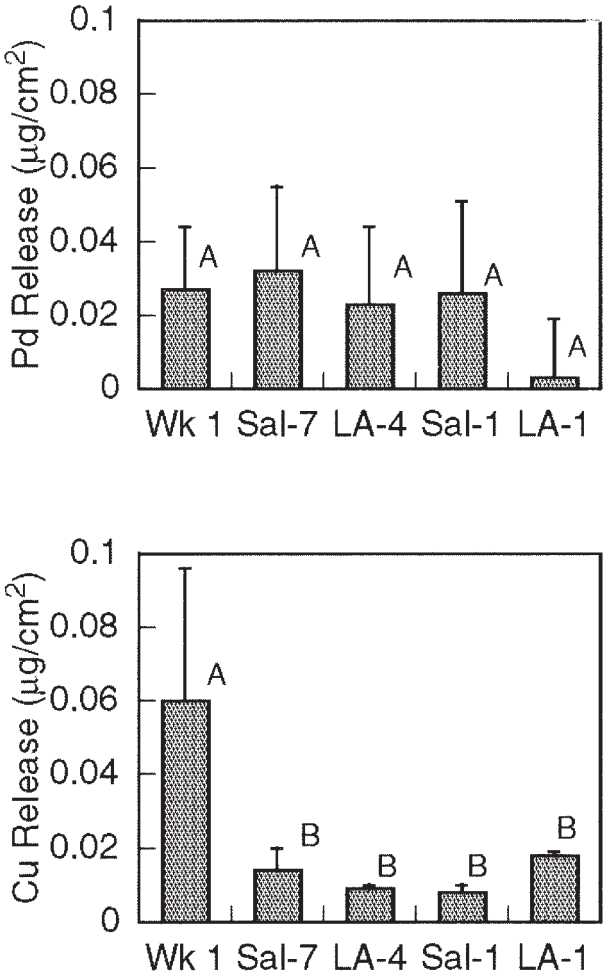

Fig. 6. Elements released from Pd-Cu-Ga noble alloy into cell-culture medium for 1 week before (column labeled Wk 1) and after 30-minute exposure to (a) saline at pH 7 (Sal-7), (b) 0.1 M lactate at pH 4 (LA-4), (c) saline at pH 1 (Sal-1), (d) or 0.1 M lactate at pH 1 (LA-1). Detection limits for Cu and Pd were 0.009 and 0.056 μg/cm^2, respectively. Error bars indicate 1 standard deviation of n = 6. Different letters adjacent to columns indicate statistical differences (ANOVA, Tukey, α=.05).

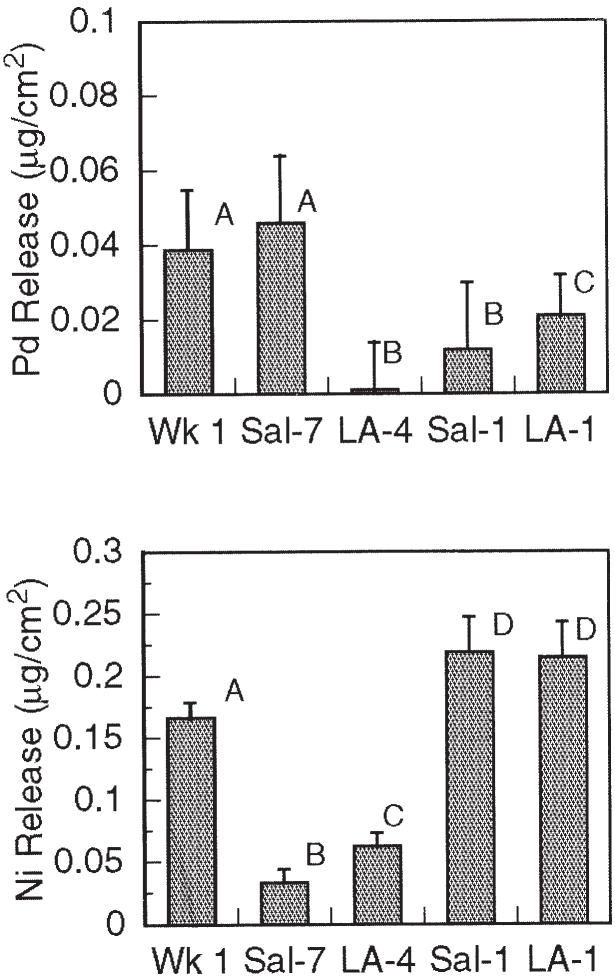

Fig. 7. Elements released from Au-Pd high-noble alloy (top) or base metal alloy (bottom) into cell-culture medium before (column labeled Wk 1) and after 30-minute exposure to (a) saline at pH 7 (Sal-7), (b) 0.1 M lactate at pH 4 (LA-4), (c) saline at pH 1 (Sal-1), or (d) 0.1 M lactate at pH 1 (LA-1). Detection limits for Pd and Ni were 0.029 and 0.005 μg/cm^2, respectively. Error bars indicate 1 standard deviation of n = 6. Different letters adjacent to columns indicate statistical differences (ANOVA, Tukey, α=.05).

1 increased Ni to levels significantly higher than those in the first week (P<.05).

Effect of increased time of exposure

When the time of treatment with lactate at pH 4 was increased from 30 to 240 minutes, the elemental release during the exposure did not increase for the Au-Pt, Pd-Cu-Ga, or Au-Pd alloys, although a slight but statistically insignificant increase for Pd was seen for the Au-Pd alloy. However, Ni release increased from 1.5 μg/cm^2 for the 30-minute exposure to over 10 μg/cm^2 for the 240-minute exposure (data not shown). In the week after the exposure to the treatment solution, the 240-minute treatment at pH 4 increased subsequent Ni release from the Ni-based alloy from 0.16 to 0.3 μg/cm^2 (Fig. 8), which was more than that observed with a 30-minute treatment at pH 1. For the other alloys, increased treatment time had no effect on element release in the second week over the 30-minute treatment.

DISCUSSION

The results of this study confirmed several previously reported observations. First, element release from dental casting alloys into a biologic medium is higher initially. This observation has been previously reported for a variety of dental casting alloys.[13,16,26] Second, the lability (tendency of elements to be released) of ele-

ments from Ni-based alloys in reduced pH solutions was confirmed, as previously reported by Covington.[23] This lability appears significant at pH of 4 or below. Covington[23] reported significant Ni release (about 2.5 μg/cm²) even at pH 6 when the extraction time was 120 days. Our study demonstrated that even brief (30 minute) exposures to these environments increased Ni release significantly.

The results demonstrated the relative stability of high-noble and noble alloys in reduced pH solutions relative to a Ni-based alloy. Although some increases in element release were observed for the high-noble (Fig. 2) and noble (Fig. 3) alloys, the increases were minor when compared with Ni release from the Ni-based alloy (Fig. 4, lower) and did not increase even when treatment was extended to 240 minutes. Furthermore, the high-noble and noble alloys only released increased elements at the relatively harsh pH 1 condition, whereas the Ni-based alloy released substantial Ni at pH 4, and yet more when treatment time was extended from 30 to 240 minutes.

High-noble and noble alloys exhibited little or no elemental release in the week after exposure to a reduced pH environment, even when pH was lowered to the severe pH 1 condition (Figs. 5 through 7). For the Au-Pt (Fig. 5) and Au-Pd (Fig. 7, top) alloys, exposure to lactate solutions decreased the release of Pd. The cause of this effect is not known, but could involve a passivation (chemical alteration that limits corrosion) of the alloy surface. Acid treatments have been used to passivate other types of alloys.[28] In contrast, when the Ni-based alloy was exposed to pH 4 or 1, the release of Ni into cell-culture medium in the subsequent week was elevated (Fig. 7, lower). When treatment time of the pH 4 solution was extended to 240 minutes, Ni release in the subsequent week was twice that observed in the first week. The lability of Ni from these alloys at a pH known to occur intraorally under plaque indicated that, clinically, the body burden of Ni may have been significantly more than observed in static tests at pH 7.

Our study demonstrated that composition of the exposing solution affected elemental release even when the pH was equivalent. For example, Cu release from the Au-Pt alloy was significantly different in saline than in lactate at pH 1 (Fig. 2). However, this effect was dependent on the element involved and the type of alloy. For the Ni-based alloy, pH appeared to be the dominating factor and composition of the solution was less important (Fig. 4, lower). The interaction between treatment solution and the alloy is undoubtedly complex. Previous studies support the idea that composition of the alloy surface is critical to elemental release behavior for high-noble, noble, and base metal alloys.[25,29] It is possible that the alloy surface also plays an important role in the interaction of a reduced pH solution, although no evidence is available to support this idea.

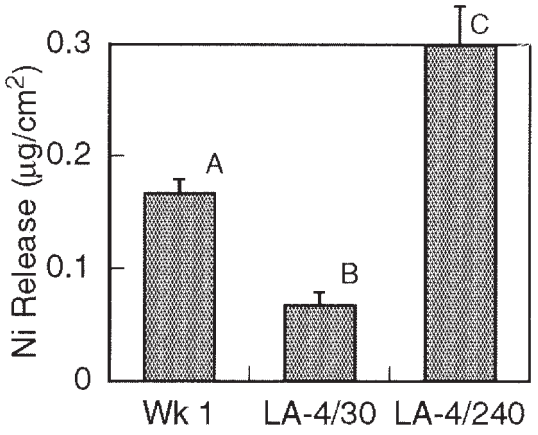

Fig. 8. Nickel released from base metal alloy into cell-culture medium before (column labeled Wk 1) and after exposure to 0.1 M lactate at pH 4 for either 30 minutes (LA-4/30) or 240 minutes (LA-4/240). Detection limit for Ni was 0.005 μg/cm². Error bars indicate 1 standard deviation of n = 6. Different letters adjacent to columns indicate statistical differences (ANOVA, Tukey, α=.05).

The dynamic nature of intraoral conditions extend beyond a simple reduction in pH. Dental alloys are subjected to a variety of chemical environments from foods and disease states. Alloys also experience mechanical disruption from occlusal forces and tooth brushing. The role of these dynamic conditions on the release of elements from alloys is virtually unknown, but it has been indicated that they are important.[30] The known adverse biologic effects of some elements such as toxicity, mutagenicity, and allergenicity is a clear rationale for understanding elemental release from these alloys.[31] Our study supports the concept that the total release of mass that an alloy contributes to the body locally and systemically must consider dynamic variables such as a temporary reduction in pH.

CONCLUSIONS

Within the limits of this study, the following conclusions were drawn:

1. High-noble and noble alloys are resistant to reduced pH environments relative to Ni-based alloys in terms of release of elements during transient (30 minutes) exposures to these environments.

2. Transient exposure of high-noble and noble elements to reduced pH does not increase release of elements once the pH is returned to pH 7. In some cases, the exposure to reduced pH decreased subsequent elemental release.

3. Transient exposure of a Ni-based alloy increased subsequent release of Ni once pH was returned to pH 7, and the effect was increased if the exposure was increased.

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