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Metal ions release influenced by acidity from arch wire when placed in artificial saliva of different pH.

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ABSTRACT

Background: Orthodontic archwires are continuously exposed to the dynamic oral environment, where variations in salivary pH can influence their corrosion behavior and subsequent metal ion release. Acidic conditions resulting from dietary habits, plaque metabolism, and carbonated beverage consumption may compromise the protective oxide layer of metallic orthodontic materials, potentially affecting their biocompatibility and clinical performance.

Aim: This study aimed to evaluate the influence of salivary acidity on the release of metal ions from orthodontic archwires immersed in artificial saliva of varying pH levels.

Materials and Methods: An in vitro experimental study was conducted using 0.016-inch nickel–titanium (NiTi) and stainless steel (SS) orthodontic archwires. Forty standardized archwire specimens were immersed in artificial saliva adjusted to four pH levels (7.0, 6.0, 5.0, and 3.5) and incubated at 37 ± 1°C for 28 days. Metal ion release was evaluated at Days 1, 7, 14, and 28 using Inductively Coupled Plasma–Optical Emission Spectroscopy (ICP-OES). Surface alterations were assessed using Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS). Statistical analysis was performed using two-way repeated-measures ANOVA and Tukey's post hoc test, with significance set at $p < 0.05$.

Results: Metal ion release increased significantly with decreasing pH and prolonged immersion duration ($p < 0.001$). The highest concentrations of nickel, chromium, iron, and titanium ions were observed in the highly acidic environment (pH 3.5). Nickel concentrations increased from 26.8 ± 2.1 µg/L on Day 1 to 68.3 ± 5.2 µg/L on Day 28, while iron exhibited the highest overall release, reaching 101.8 ± 6.6 µg/L at Day 28. SEM analysis demonstrated progressive surface deterioration characterized by pitting corrosion, microcracks, and surface roughening under acidic conditions. EDS findings confirmed elemental depletion of metallic constituents and increased surface oxidation following immersion.

Conclusion: Salivary acidity significantly influences the corrosion behavior and metal ion release of orthodontic archwires. Acidic environments accelerate degradation of the protective oxide layer, resulting in increased metal dissolution and surface deterioration. Although the detected ion concentrations remained within biologically acceptable limits, prolonged exposure to acidic conditions may have clinical implications, particularly in patients with metal hypersensitivity. Maintenance of a neutral oral environment may help reduce corrosion and improve the long-term biocompatibility of orthodontic appliances...

Keywords: Orthodontic archwires; Corrosion; Salivary pH; Metal ion release; Nickel–titanium; Stainless steel; ICP-OES; Scanning electron microscopy; Biocompatibility; Artificial saliva.

INTRODUCTION

Orthodontic archwires are continuously exposed to the oral environment, where fluctuations in pH, temperature, enzymatic activity, and microbial metabolites can influence their corrosion behavior. Corrosion is an electrochemical process resulting in the degradation of metallic materials and the release of metal ions into the surrounding environment. The oral cavity presents a particularly challenging environment because dietary acids, plaque metabolism, and fluoride-containing products may lower salivary pH and accelerate corrosion.

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The most commonly used orthodontic archwires are manufactured from stainless steel (SS), nickel–titanium (NiTi), beta-titanium (TMA), and cobalt–chromium alloys. These materials contain varying proportions of nickel, chromium, iron, titanium, molybdenum, and cobalt, which may be released into saliva through corrosion processes. Released ions have been associated with allergic reactions, cytotoxicity, mucosal irritation, and alterations in local tissue responses, particularly in susceptible individuals.

Previous investigations have demonstrated that acidic conditions significantly increase ion release from orthodontic appliances. Lower pH values destabilize the protective oxide layer present on metallic surfaces, thereby enhancing corrosion rates and increasing concentrations of nickel and chromium ions in artificial saliva

Although several studies have examined metal ion release from orthodontic materials, comparative evaluation of archwire corrosion under different salivary pH conditions remains clinically relevant due to frequent exposure of orthodontic patients to acidic beverages, soft drinks, and cariogenic biofilms. Therefore, the present study aimed to evaluate the influence of salivary acidity on the release of metal ions from orthodontic archwires immersed in artificial saliva of varying pH levels.

Materials and Methodology

This in vitro experimental study was conducted to evaluate the influence of salivary acidity on metal ion release from orthodontic archwires. Commercially available orthodontic archwires, including 0.016-inch nickel–titanium (NiTi) and 0.016-inch stainless steel (SS) archwires obtained from the same manufacturer batch, were used in the study. The archwires were cut into standardized 20-mm segments using a tungsten carbide cutter to ensure uniform surface area exposure. Prior to experimentation, all specimens were ultrasonically cleaned in deionized water for 10 minutes to remove surface contaminants, rinsed thoroughly, and dried under sterile conditions. Similar specimen preparation protocols have been employed in previous corrosion studies evaluating orthodontic materials.(1–3)

Artificial saliva was prepared according to the Fusayama–Meyer formulation, consisting of sodium chloride (0.4 g/L), potassium chloride (0.4 g/L), calcium chloride dihydrate (0.795 g/L), sodium dihydrogen phosphate dihydrate (0.69 g/L), sodium sulfide nonahydrate (0.005 g/L), and urea (1.0 g/L) dissolved in distilled water.(4) The pH of the artificial saliva was adjusted using 0.1 M hydrochloric acid (HCl) and 0.1 M sodium hydroxide (NaOH) to obtain four experimental conditions: pH 7.0 (neutral), pH 6.0 (slightly acidic), pH 5.0 (moderately acidic), and pH 3.5 (highly acidic). These pH levels were selected to simulate normal salivary conditions and the acidic oral environment that may result from dietary acids, plaque accumulation, and frequent consumption of carbonated beverages.(1,5)

A total of forty archwire specimens were randomly allocated into four groups (n = 10 per group) according to the pH of the immersion medium. Each specimen was placed individually in sterile polypropylene containers containing 20 mL of artificial saliva and incubated at 37 ± 1°C to simulate intraoral conditions. The immersion media were renewed every seven days to maintain pH stability and prevent ion saturation. Samples were collected at four predetermined intervals: Day 1, Day 7, Day 14, and Day 28. At each time point, aliquots of the immersion solution were collected and stored in acid-washed polyethylene tubes at 4°C until analysis.(2,6)

Quantitative analysis of released metal ions was performed using Inductively Coupled Plasma–Optical Emission Spectroscopy (ICP-OES) due to its high sensitivity and accuracy in trace metal detection. The concentrations of nickel (Ni), chromium (Cr), iron (Fe), and titanium (Ti) ions were measured and expressed in micrograms per liter (µg/L). Calibration curves were established using certified standard solutions before each analytical run to ensure measurement reliability.(7)

To evaluate surface alterations resulting from corrosion, representative archwire specimens from each group were examined before and after immersion using Scanning Electron Microscopy (SEM). Surface morphology, pitting, cracks, and evidence of corrosion were documented at multiple magnifications. In addition, Energy Dispersive X-ray Spectroscopy (EDS) was performed to assess elemental surface composition and detect changes associated with metal dissolution.(3,8)

Statistical analysis was conducted using SPSS software version 29.0 (IBM Corp., Armonk, NY, USA). Data normality was assessed using the Shapiro–Wilk test. Descriptive statistics were expressed as mean ± standard deviation. The effects of pH and immersion time on metal ion release were analyzed using two-way repeated-measures analysis of variance (ANOVA), followed by Tukey's post hoc test for pairwise comparisons. A p-value less than 0.05 was considered statistically significant.

DISCUSSION

The present study demonstrated that decreasing salivary pH significantly increased metal ion release from orthodontic archwires. The highest concentrations of nickel, chromium, and iron were observed in specimens immersed in artificial saliva at pH 3.5.These findings support previous investigations showing that acidic environments compromise the passive oxide layer protecting orthodontic alloys. Once this passive film is disrupted, electrochemical corrosion accelerates, resulting in increased ion liberation.

Nickel exhibited the greatest increase among the measured ions. This observation is clinically relevant because nickel hypersensitivity affects a considerable proportion of the population and may manifest as gingival inflammation, mucosal erythema, or contact dermatitis. The progressive increase in ion release over the 28-day period suggests cumulative corrosion behavior. Similar temporal patterns have been reported in previous in vitro studies investigating orthodontic alloys exposed to acidic media.

SEM observations revealed extensive surface deterioration under acidic conditions. Corrosion pits and surface irregularities observed after immersion at pH 3.5 indicate localized breakdown of the protective oxide layer, thereby facilitating further ion release.Although the released metal concentrations remained below established toxic thresholds, chronic exposure and cumulative biological effects cannot be disregarded, particularly in patients with known metal hypersensitivity.

Results

A total of 40 orthodontic archwire specimens were evaluated for metal ion release under varying pH conditions over a 28-day immersion period. Quantitative analysis using ICP-OES revealed that both salivary pH and immersion duration significantly influenced the release of metallic ions from the archwires. Overall, metal ion release increased progressively with decreasing pH and increasing exposure time, indicating enhanced corrosion activity in acidic environments.

Nickel Ion Release

Nickel ion release demonstrated a clear inverse relationship with salivary pH (Table 1). At neutral pH (7.0), the mean nickel concentration increased gradually from 5.2 ± 0.8 μg/L on Day 1 to 12.1 ± 1.5 μg/L on Day 28. In contrast, specimens immersed in acidic artificial saliva exhibited significantly greater nickel release. At pH 6.0, the concentration increased from 8.5 ± 1.0 μg/L to 22.4 ± 2.3 μg/L, while at pH 5.0 it increased from 15.6 ± 1.7 μg/L to 39.7 ± 3.2 μg/L during the same period. The highest nickel release was observed in the pH 3.5 group, where concentrations rose from 26.8 ± 2.1 μg/L on Day 1 to 68.3 ± 5.2 μg/L by Day 28 (Table 1). Statistical analysis demonstrated significant differences among all pH groups ($p < 0.001$), confirming that increased acidity accelerated nickel dissolution from orthodontic archwires.

Chromium Ion Release

Chromium ion release followed a pattern similar to nickel release (Table 2). At pH 7.0, chromium concentrations remained relatively low, increasing from 1.8 ± 0.4 μg/L on Day 1 to 4.6 ± 0.9 μg/L on Day 28. As the pH decreased, chromium release increased substantially. The pH 6.0 group showed chromium concentrations ranging from 3.1 ± 0.5 μg/L to 9.1 ± 1.0 μg/L, whereas the pH 5.0 group exhibited concentrations between 6.7 ± 0.8 μg/L and 18.6 ± 1.8 μg/L. The highest chromium release was recorded in the pH 3.5 group, with concentrations increasing from 11.4 ± 1.2 μg/L to 34.7 ± 2.9 μg/L over the 28-day period (Table 2). The differences among the groups were statistically significant ($p < 0.001$).

Iron Ion Release

Among all measured ions, iron exhibited the highest absolute concentrations throughout the study (Table 3). In the neutral pH group, iron concentrations increased from 12.3 ± 1.1 μg/L on Day 1 to 24.1 ± 2.0 μg/L on Day 28. At pH 6.0, concentrations increased from 18.6 ± 1.5 μg/L to 40.4 ± 3.3 μg/L, while the pH 5.0 group showed values ranging from 29.4 ± 2.3 μg/L to 68.7 ± 5.0 μg/L. The greatest iron release occurred at pH 3.5, where concentrations increased from 45.8 ± 3.2 μg/L on Day 1 to 101.8 ± 6.6 μg/L on Day 28 (Table 3). The results indicate that acidic conditions markedly increased corrosion of iron-containing orthodontic alloys.

Titanium Ion Release

Titanium release was comparatively lower than that of nickel, chromium, and iron. Nevertheless, a significant increase in titanium ion concentration was observed with decreasing pH and prolonged immersion. At pH 7.0, titanium concentrations increased from 0.9 ± 0.2 μg/L to 2.5 ± 0.4 μg/L over 28 days. The highest titanium release was observed in the pH 3.5 group, reaching 12.4 ± 1.1 μg/L by Day 28. These findings suggest that titanium-containing alloys possess superior corrosion resistance when compared with nickel- and iron-containing orthodontic materials.

Effect of Immersion Duration

Regardless of pH, all groups exhibited a time-dependent increase in metal ion release. The highest increase was observed between Day 14 and Day 28, suggesting cumulative corrosion with prolonged exposure. The cumulative release of nickel, chromium, and iron after 28 days was approximately five- to six-fold higher in the pH 3.5 group than in the neutral pH group (Tables 1–3). These findings indicate that both acidity and exposure duration play critical roles in determining corrosion behavior.

Surface Morphological Analysis

Scanning Electron Microscopy (SEM) examination revealed distinct differences in surface morphology before and after immersion (Figure 2). Baseline specimens exhibited relatively smooth surfaces with minimal manufacturing defects. Following immersion in neutral artificial saliva, only minor surface irregularities were observed. However, specimens exposed to acidic media demonstrated progressive surface deterioration characterized by pitting corrosion, microcracks, surface roughening, and localized material loss. The most severe surface degradation was observed in specimens immersed in artificial saliva at pH 3.5 for 28 days (Figure 2).

Energy Dispersive X-ray Spectroscopy Analysis

EDS analysis demonstrated a reduction in surface concentrations of nickel, chromium, and iron following immersion, particularly under acidic conditions. Concurrently, an increase in oxygen content was observed, suggesting oxidation and formation of corrosion products. These findings corroborated the ICP-OES data and confirmed the occurrence of corrosion-induced metal dissolution in acidic environments.

Inferential Statistical Analysis

Two-way repeated-measures ANOVA demonstrated a highly significant effect of salivary pH on metal ion release ($F = 85.62$, $p < 0.001$), as well as a significant effect of immersion duration ($F = 73.14$, $p < 0.001$) (Table 4). Furthermore, a significant interaction effect between pH and immersion time was identified ($F = 29.76$, $p < 0.001$), indicating that the influence of acidity became progressively more pronounced with increasing exposure duration. Tukey's post hoc analysis revealed significant differences between all pH groups ($p < 0.05$), confirming that each reduction in pH resulted in a measurable increase in metal ion release.

Table 1. Mean Nickel Ion Release (µg/L)

Nickel release increased significantly with decreasing pH and increasing immersion time ($p < 0.001$), consistent with previous corrosion studies.

Source	**F-value**	**p-value**
pH	85.62	<0.001
Time	73.14	<0.001
pH × Time	29.76	<0.001

Table 2. Mean Chromium Ion Release (µg/L)

pH	**Day 1**	**Day 7**	**Day 14**	**Day 28**
7.0	1.8 ± 0.4	2.9 ± 0.6	3.8 ± 0.8	4.6 ± 0.9
6.0	3.1 ± 0.5	5.4 ± 0.7	7.2 ± 0.9	9.1 ± 1.0
5.0	6.7 ± 0.8	10.5 ± 1.2	14.8 ± 1.5	18.6 ± 1.8
3.5	11.4 ± 1.2	19.2 ± 1.8	26.3 ± 2.4	34.7 ± 2.9

Table 3. Mean Iron Ion Release (µg/L)

pH	Day 1	Day 7	Day 14	Day 28
7.0	12.3 ± 1.1	16.7 ± 1.4	20.4 ± 1.7	24.1 ± 2.0
6.0	18.6 ± 1.5	26.2 ± 2.1	33.5 ± 2.8	40.4 ± 3.3
5.0	29.4 ± 2.3	41.6 ± 3.1	55.2 ± 4.1	68.7 ± 5.0
3.5	45.8 ± 3.2	63.1 ± 4.7	82.4 ± 5.9	101.8 ± 6.6

Table 4. Two-Way ANOVA

Source	F-value	p-value
pH	85.62	<0.001
Time	73.14	<0.001
pH × Time	29.76	<0.001

CONCLUSION

Within the limitations of this in vitro study, it can be concluded that salivary acidity has a significant influence on the corrosion behavior and metal ion release of orthodontic archwires. A progressive increase in the release of nickel, chromium, iron, and titanium ions was observed with decreasing pH and increasing immersion duration. Archwires immersed in highly acidic artificial saliva (pH 3.5) exhibited the greatest metal ion release and the most pronounced surface degradation, whereas those maintained in neutral artificial saliva (pH 7.0) demonstrated the lowest corrosion activity. The findings indicate that acidic oral environments can compromise the protective oxide layer of orthodontic alloys, thereby accelerating corrosion and enhancing metal ion dissolution. Although the measured ion concentrations remained within biologically acceptable limits, prolonged exposure to acidic conditions may increase the risk of adverse biological responses, particularly in individuals with metal hypersensitivity. Therefore, maintenance of a neutral oral pH through appropriate dietary habits and oral hygiene measures may help minimize corrosion of orthodontic appliances and improve their long-term biocompatibility and clinical performance. Future studies incorporating different archwire alloys, fluoride-containing media, and in vivo conditions are recommended to further elucidate the clinical implications of orthodontic material corrosion...

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