Dentistry 3000

Vol 14, No 2 (2026)
ISSN 2167-8677 (online)
DOI 10.5195/d3000/2026.1541

Effect of Hyperglycemic Artificial Saliva on Corrosion of and Metal Ion Resease from Archwires

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Abstract

Alterations in the intraoral conditions, especially raised glucose concentrations, may compromise the surface integrity of archwire materials enhancing metallic ion elution. The purpose of the present in vitro study was to assess the effect of artificial saliva in high glucose conditions on corrosion behavior, surface deterioration, color change and release of metal ions from various rectangular orthodontic archwires. Three commercially available orthodontic archwire types were compared: stainless steel (SS), nickel–titanium (NiTi) and beta-titanium (β-Ti). A total of 180 wire specimens were equally distributed into four immersion media: glucose-free artificial saliva (control), artificial saliva containing 10 mmol/L glucose, 20 mmol/L glucose, and 30 mmol/L glucose. Specimens were immersed for 28 days at 37°C. Surface morphology was evaluated using scanning electron microscopy (SEM), elemental composition by energy-dispersive X-ray spectroscopy (EDX), surface roughness using atomic force microscopy (AFM), color change (ΔE) using a spectrophotometer, and metal ion release (Ni, Cr, Fe, and Ti) using inductively coupled plasma–mass spectrometry (ICP-MS). Statistical analysis was performed using one-way ANOVA with statistical significance established at $p < 0.05$. Glucose-enriched artificial saliva significantly increased surface roughness in all orthodontic archwires across glucose concentrations ($p < 0.001$). The values of surface roughness gradually increased as the glucose concentration increased. The Ni ion release from NiTi wires was 0.82 ± 0.14 μg/L in the control group and 3.96 ± 0.42 μg/L after 28 days of immersion in the 30 mmol/L glucose enriched artificial saliva ($p < 0.001$). The chromium and iron releases were also significantly increased from stainless steel wires in high-glucose conditions, while the Beta-Ti wires showed the fewest corrosion-related changes with minimal release of titanium ions. Groups with glucose enrichement had greater color change (ΔE). Beta-titanium samples exhibited lower surface degradation. Glucose-enriched artificial saliva increased surface roughness, release of metal ions, surface degradation and color change of orthodontic archwires in a concentration-dependent manner. Beta-titanium archwires showed fewer corrosion changes than nickel–titanium and stainless-steel wires.

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Citation: Taha SS et al. (2026) Effect of Hyperhlycemic Artificial Saliva on Corrosion of and Metal Ion Release from Archwires. Dentistry 3000. 1:a001 doi:10.5195/d3000.2026.1541
Received: August 4, 2026
Accepted: August 5, 2026
Published: August 18, 2026

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Introduction

The oral environment is considered a sophisticated electrochemical medium in which saliva serves as electrolytes around metal based orthodontic devices. Artificial saliva medium is extensively employed as a soaking medium to mimic intraoral environment in laboratory corrosion investigations. The corrosion response of orthodontic arch wire materials perhaps affected by elemental composition, surface preparation, period of exposure, and physiochemical property of saliva especially PH. Earlier investigations have established that nickel-titanium and stainless-steel orthodontic wire materials may be subjected to surface damage post exposure to artificial salivary medium. Increasingly acidic circumstances may diminish the persistence of the surface oxide

Vol 14, No 2 (2026) DOI 10.5195/d3000/2026.1541

protective film developed on arch wire exterior surface, consequently raising surface topographic roughness, localized corrosive attack, vulnerability to additional subsequent degradation [1–3].

Diabetes mellitus is one of the most common chronic metabolic diseases in the world and consists of chronic hyperglycemia due to a relative or absolute decrease in the production of insulin or an increase in insulin resistance. Numerous physiological systems are impaired by chronic hyperglycemia, such as the oral cavity, which has been shown to have decreased salivary flow, elevated salivary glucose levels, impaired buffering capacity, oxidative stress, and increased risk of periodontal disease and oral infections. These biochemical changes can also impact the electrochemical properties of the dental metallic materials, possibly enhancing the corrosion processes [4].

It has been shown in several investigations that increasing glucose levels increases oxidative reactions and instabilities in electrochemical processes of metallic biomaterials. In hyperglycemic situations, passive oxide layers which protect orthodontic alloys from degradation can be affected. When this passive layer is broken down, corrosion takes place much faster, leading to the release of more ions and deterioration of surface. These changes can affect the mechanical properties and esthetic appearance of orthodontic archwires throughout the course of their clinical use in the long term [5].

Despite the differences in the metallurgical composition and corrosion resistance profile of the archwires of stainless steel, nickel–titanium (NiTi), and beta-titanium (TMA), their performance in the oral environment of a diabetic patient has not been well understood. The corrosion resistance of stainless-steel alloys is mainly attributed to the passive film, which is rich in chromium (less common NiTi), whilst NiTi alloys rely on the passive film of $TiO2$ layers, that may be unstable in chemically aggressive conditions. Under hyperglycemic circumstances, the corrosion resistance of beta-Ti alloys is generally better due to the highly stable nature of their oxide surface; however, comparative evidence is not widely available [5].

The purpose of the present in vitro study was to assess the effect of artificial saliva in high-glucose conditions on corrosion behavior, surface deterioration, color change and release of metal ions from various rectangular orthodontic archwires.

Material and Methods

Study Design

This study is in vitro experimental study which investigate the corrosion behavior, metal ion release, surface roughness and color changes of three orthodontic archwire alloys in the presence of high-glucose artificial saliva. A standardized protocol was used for the experiments to ensure reproducibility and to reduce the confounding variables.

Three commonly used orthodontic archwire alloys were used.: Stainless steel (SS), Nickel–titanium (NiTi), and Beta-titanium (TMA™). The archwires were all rectangular in shape 0.016×0.022 inch and were manufactured from the same batch (Ormco Corporation®, USA). Each wire segment was cut into 20 mm pieces (from flat non-curved end archwire) with a sterile orthodontic wire cutter before starting testing. The cut surfaces were first polished by silicon carbide abrasive papers and then ultrasonically cleaned in distilled water for 10 minutes to eliminate the surface contamination [6]. Sample size was determined by G*Power software version 3.1 for two-way ANOVA depend on interaction effect between material of archwire and glucose concertation with an effect size of f=0.28 and a significance level of α=0.05, and the statistical power of 80% yielding the total sample size which was 180 specimens, the specimens were equally distributed into three experimental groups according to three archwire materials (SS, NiTi, and β-Ti) each group contained 60 specimens which was divided into four subgroups according to four immersion media, Each subgroup contained fifteen specimens (n = 15).

Artificial saliva was prepared according to a modified Fusayama formulation containing:

Component	Concentration (g/L)
NaCl	0.4
KCl	0.4
$CaCl_2·2H_2O$	0.795
$NaH_2PO_4·2H_2O$	0.69
$Na_2S·9H_2O$	0.005
Urea	1.0

A solution with pH 6.8 ± 0.1 was obtained by adjusting the solution with 0.1 M NaOH or 0.1 M HCl. Four immersion solutions were prepared: Group I glucose-free artificial saliva (Control), Group II artificial saliva with 10mmol/L glucose, Group III 20mmol/L glucose containing artificial saliva, and Group IV artificial Saliva with 30mmol/L glucose. The concentrations of glucose were chosen to represent rising salivary glucose level that is seen in DM patients.

The individual specimens were placed in 20mL of each solution in sterile polypropylene tubes. The tubes were placed in the incubator at 37°C, relative humidity was 100%, and immersion period was 28 days. The immersion solutions were collected separately and replaced every 48 hours to maintain chemical stability and prevent bacterial contamination.

Surface roughness (Ra, µm) was measured using an Atomic Force Microscope (AFM) operating in tapping mode with scan area of $10*10µm^2$. Three measurements were obtained from different regions of each specimen [7]. The average value was recorded.

Surface topography was examined using Scanning Electron Microscopy (SEM) [8]. Representative specimens from each experimental group were sputter-coated with gold before imaging. Micrographs were obtained at magnifications ranging from 500× to 5000× to evaluate pitting corrosion, surface cracks, irregularities, and localized corrosion defects.

The elemental composition of the wire surfaces was determined using Energy Dispersive X-ray Spectroscopy (EDX) attached to the SEM [9]. The percentages of nickel, chromium, iron, titanium, and oxygen were quantified before and after immersion.

The concentrations of released metallic ions were determined using Inductively Coupled Plasma–Mass Spectrometry (ICP-MS) [10]. Nickel (Ni), Chromium (Cr), Iron (Fe), and Titanium (Ti) ions were measured. Ion concentrations were expressed as µg/L. Calibration standards were prepared before each analytical session according to the manufacturer's recommendations.

Color changes were measured using a spectrophotometer according to the CIE L*a*b* color system. Color difference (ΔE) was calculated using the equation [11]:

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$$

Three measurements were performed for each specimen, and the mean value was calculated. The corrosion behavior was assessed using surface roughness changes, SEM observations, EDX elemental alterations, metal ion release, and color alteration. These complementary approaches gave a detailed characterization of the corrosion process under glycemic conditions.

The statistical analysis was conducted using IBM SPSS Statistics (Version 29.0; IBM Corp., Armonk, NY, USA) was used to analyze the data. Test of normality for the distribution of data was performed with Shapiro-Wilk test. Comparisons among experimental groups were performed using One-way analysis of variance (ANOVA). Statistical significance was established at $p < 0.05$.

Results

Surface Roughness

The results of one-way ANOVA showed that there was a significant increase in surface roughness after immersion in artificial saliva with various glucose concentrations; the extent of this increase varied with the concentration of glucose. Progressive increase in the roughness value was observed in SS and NiTi archwires while lower changes were observed following the experimental period for β-Ti archwires. After 28 days of immersion in artificial saliva containing 30 mmol/L glucose, stainless steel archwires demonstrated the highest surface roughness (0.79 ± 0.05 µm), followed by NiTi (0.72 ± 0.04 µm), while beta-titanium maintained lower values (0.41 ± 0.03 µm) ($p < 0.001$).

Metal Ion Release

The released metallic ion concentrations were found to be higher with increasing glucose concentration. Under hyperglycemic conditions, higher Ni release from NiTi archwires was observed. The release of Fe and Cr from the stainless steel was also higher in comparison to Ti release from NiTi, and Ti release from Beta-titanium was relatively low after 28 days of immersion.

Color Stability

Color changes were found in all types of archwires after immersion. Color changes were measured for all archwires after immersion. It was observed that the color change (ΔE) increased with the concentration of glucose. Samples of stainless steel that were immersed in 30 mmol/L glucose showed the highest color shift. The color change was more pronounced in the stainless-steel samples that were immersed in 30 mmol/L glucose. The color stability of Beta-titanium was greater than that of other materials.

SEM Evaluation

All the control specimens were found to be smooth and homogeneous under scanning electron microscopy. The specimens were immersed in the hyperglycemic saliva, and subsequently exhibited a corrosion defect on the surface. Minor irregularities and shallow pits were seen at a glucose concentration of 10 mmol/L. At the intermediate glucose concentration (20 mmol/L), more severe corrosion was observed: localized pitting and disruption of the passive oxide film were seen. When the glucose concentration reached 30 mmol/L, corrosion was observed in stainless steel and NiTi archwires. The bull's-eye defects and irregular grooves/cracks on the surface, as well as general degradation were often observed. Shallow defects were seen in isolated areas on the surface of Beta-titanium archwires.

EDX Analysis

The chromium concentration decreased in the hyperglycemic saliva on the stainless-steel surface and the NiTi surface showed a decrease in nickel concentration in the hyperglycemic saliva. Meanwhile, the oxygen content increased, indicative of increased oxide formation associated with corrosion. The elemental changes in beta-titanium were minimal when compared with the other alloys.

Overall Corrosion Assessment

Highest corrosion resistance was observed in Beta-titanium and greatest corrosion-related changes were observed in stainless steel with the greatest colour change. Nickel–titanium had the greatest nickel ion release.

Discussion

The present study revealed that the increasing glucose concentrations in artificial saliva had a significant effect on the corrosion behavior of orthodontic archwires, and on the surface roughness, metal ion release and colour stability. The present observations indicate that the hypothesis that an association exists between increased glucose and changes in the electrochemical conditions of the artificial saliva and hence the susceptibility to corrosion of metallic orthodontic materials is plausible. The present results indicate that high glucose concentrations may also be a potential factor that influences the material performance of the orthodontic archwires, under conditions associated with poorly controlled diabetes mellitus (DM), while the acidic pH, fluoride-containing agents and mouthwashes are considered corrosive factors [12-14]. All types of archwires showed greater roughness with higher glucose concentrations, differences were statistically significant. This finding is likely due to the concentration-dependent disturbance of the protective layer of passive oxide that develops naturally on metallic orthodontic alloys.

Stainless steel archwires showed marked surface deterioration with iron and chromium ion release among the materials investigated. The main reason for the corrosion resistance of stainless steel is the passive oxide film with a high chromium content, but the passive film can be destabilized by prolonged exposure to an aggressive electrolyte thereby promoting electrochemical dissolution. Previous laboratory studies showed that stainless-steel orthodontic wires degrade over time when immersed in artificial saliva under corrosive conditions, resulting in greater susceptibility to pitting corrosion [15-17].

Nickel–titanium archwires had intermediate corrosion resistance. These observations have significant biological implications as nickel is one of the most common causes of hypersensitivity associated with orthodontic materials. Consistent with the literature, nickel ion release has been found to depend on the alloy composition, immersion medium, polishing, exposure time, exposure temperature and integrity of the titanium oxide passive layer [13,17].

Beta-titanium archwires had the greatest corrosion resistance and minimal surface degradation. The main reason for their superior performance is thought to be the formation of a dense layer of titanium dioxide (TiO_2), which acts to effectively protect the underlying alloy from electrochemical attack. Surface deterioration and the release of titanium ions were found to be lower than those in NiTi archwires in every case. The lower surface deterioration and reduced Ti ion release were supported by the surface observations, elemental changes analysis and ion release measurements compared with NiTi archwires. The same finding has been observed in previous studies that have investigated titanium-based orthodontic alloys under simulated oral conditions [13].

Increased glucose levels can lead to increased corrosivity and to electrochemical reactions taking place at the metal surface, which can cause dissolution of nickel, chromium and iron. These findings complement the results of previous electrochemical research showing that the corrosion and release of ions increase as a function of immersion time in simulated oral fluids [13,15,16].

Conclusion

In this in vitro study, within its limitations, the corrosion-related changes of orthodontic archwires were greater in glucose-enriched artificial saliva. The surface roughness increased, metal ion release increased, surface degradation worsened, and the color change of the samples increased with increasing glucose concentration compared with glucose-free artificial saliva. Beta-titanium archwires showed higher corrosion resistance, minimal surface degradation, and lower titanium ion release. In vivo studies are recommended to validate these results in the oral environment and to evaluate how oral biofilm and mechanical loading of orthodontic archwires interact in a high-glucose environment.

Study Limitations

In the interpretation of the results of the present study, the following limitations were identified; the complexity of the human oral environment was not fully mimicked; there were no salivary proteins, enzymes, immune components or oral microorganisms present in the artificial saliva that could affect the corrosion behavior. No simulation of dynamic intraoral factors such as mastication, temperature changes, pH cycling or mechanical loading was conducted. The study was conducted on isolated archwire samples, rather than on actual complete orthodontic systems, as galvanic interaction between brackets, ligatures, and archwires could influence corrosion patterns. There were only three glucose concentrations and a single immersion period of 28 days, so duration -related changes could not be evaluated. The study only considered the corrosion behavior, the surface characteristics, color changes and the release of metal ions, and did not assess

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changes in the mechanical properties (tensile strength, elastic modulus, frictional resistance).

Other biological responses such as cytotoxicity, inflammatory biomarkers and changes in cellular oxidative stress were not examined and should be considered in future studies. To validate the findings of this study, further research is recommended, including long-term in vivo studies with patients with diabetes, investigation of various orthodontic alloys and coatings, incorporation of oral biofilm models, and the combined effects of hyperglycemia, acidic conditions, fluoride exposure, and cyclic mechanical loading on orthodontic material degradation.

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Table 1. One-way ANOVA for surface roughness (Ra, μm) of orthodontic archwires post 28-day immersion period.

Archwire type	Control (glucose-free artificial saliva)	10 mmol/L glucose	20 mmol/L glucose	30 mmol/L glucose	p-value
Stainless Steel	0.29 ± 0.02	0.45 ± 0.03	0.61 ± 0.04	0.79 ± 0.05	<0.001
NiTi	0.31 ± 0.03	0.43 ± 0.03	0.58 ± 0.04	0.72 ± 0.04	<0.001
Beta-Titanium	0.26 ± 0.02	0.31 ± 0.02	0.36 ± 0.03	0.41 ± 0.03	<0.001

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Table 2. One-way ANOVA for the released metallic ions(µg/L) after 28-day immersion period.

Archwire type	Released metallic ions	Control (glucose-free artificial saliva)	10 mmol/L glucose	20 mmol/L glucose	30 mmol/L glucose	p-value
Stainless Steel	Iron	1.84 ±0.21	2.71 ±0.26	3.94 ±0.35	5.83 ±0.42	<0.001
Stainless Steel	Chromium	0.62 ±0.08	1.14 ±0.12	1.78 ±0.15	2.69 ±0.18	<0.001
NiTi	Nickel	0.82 ±0.14	1.67 ±0.2	2.81 ±0.28	3.96 ±0.42	<0.001
NiTi	Titanium	0.39 ±0.05	0.46 ±0.06	0.53 ±0.07	0.68 ±0.08	<0.001
Beta-Titanium	Titanium	0.24 ±0.04	0.29 ±0.05	0.33 ±0.05	0.41 ±0.05	<0.001

Table 3. Color changes (ΔE) of archwires after 28-day immersion period using one-way ANOVA

Archwire type	Control (glucose-free artificial saliva)	10 mmol/L glucose	20 mmol/L glucose	30 mmol/L glucose	p-value
Stainless Steel	0.62 ±0.11	1.38 ±0.17	2.34 ±0.21	3.86 ±0.29	<0.001
NiTi	0.59 ±0.1	1.26 ±0.16	2.07 ±0.18	3.21 ±0.24	<0.001
Beta-Titanium	0.43 ±0.08	0.76 ±0.1	1.08 ±0.13	1.64 ±0.17	<0.001