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

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Abstract

The purpose of the present in vitro study was to assess the effect of the artificial saliva in hyperglycemic condition on corrosion behavior, surface deterioration, color change and release of metal ions from various orthodontic archwires. Three commercially available orthodontic archwire types were compared: stainless steel (SS), nickel–titanium (NiTi) and beta-titanium (TMA). A total of 180 wire specimens were randomly allocated into four immersion media: conventional artificial saliva (control), artificial saliva containing 10 mmol/L glucose, 20 mmol/L glucose, and 30 mmol/L glucose. Specimens were immersed for 7, 14, and 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 stability using a spectrophotometer (ΔE), 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 followed by Tukey's post hoc test, with statistical significance established at $p < 0.05$. Hyperglycemic artificial saliva significantly increased corrosion-related surface defects in all orthodontic archwires compared with the control group ($p < 0.001$). The degree of surface roughness gradually increased as the glucose concentration and immersion time increased. The amount of Ni ion released 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 submersion in the 30 mmol/L glucose saliva ($p < 0.001$). The chromium and iron release were also seen to be significant in stainless steel wires in hyperglycemic condition, while the beta-Ti wires showed the maximum corrosion resistance with minimal amount of release of titanium ions. Groups with hyperglycemia had significantly higher color alteration (ΔE), especially for the long-term exposure. Performing SEM on the stainless steel and NiTi samples revealed a lot of pits and surface irregularities, and the surface of the TMA samples remained comparatively smooth. Hyperglycemic artificial saliva has significantly effects on the corrosion, number of metallic ions released, surface integrity and colour stability of orthodontic archwires in a concentration dependent manner. Beta-titanium archwires were found to be more corrosion resistant than nickel–titanium and stainless-steel wires. Based on these results, it can be concluded that in cases of diabetes mellitus, which is not well controlled, it is advisable to carefully consider material selection and closely monitor patients during orthodontic treatment to reduce the risk of corrosion-related complications.

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Introduction

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 [1].

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 [2].

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 [2].

Material and Methods

Study Design

The objective of this study was to investigate the corrosion behavior, metal ion release, surface roughness and color changes of three orthodontic archwire alloys in the presence of hyperglycemic artificial saliva in vitro as an experimental study. A standardized protocol was used for the experiments to ensure reproducibility and to reduce the confounding variables. Three popular orthodontic archwire alloys were used: Stainless steel (SS), Nickel–titanium (NiTi), and Beta-titanium (TMA).

The archwires were all rectangular in shape and were manufactured from the same batch, ensuring a minimum variation in material. Each wire segment was cut into 20 mm pieces 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. 180 specimens were prepared altogether. The specimens were equally distributed according to three archwire materials (SS, NiTi, and TMA), four immersion media, and three immersion periods. Each subgroup contained five specimens (n = 5).

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

Component	Concentration (g/L)
NaCl	0.4
KCl	0.4
$CaCl_2 \cdot 2H_2O$	0.795
$NaH_2PO_4 \cdot 2H_2O$	0.69
$Na_2S \cdot 9H_2O$	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 solutions for immersion were made: Group I (Control, hypoglycoside artificial saliva), Group II, artificial saliva with 10mmol/L glucose, Group III, 20mmol/L glucose containing artificial saliva, and Group IV, synthetic Saliva with 30mmol/L glucose. The concentrations of glucose were chosen to represent rising salivary hyperglycaemia 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%. Immersion periods were 7, 14, and 28 days.

The immersion solutions were 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. Three measurements were obtained from different regions of each specimen. The average value was recorded.

Surface topography was examined using Scanning Electron Microscopy (SEM). 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. 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). 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:

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

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 visual color alteration. These complementary approaches gave a detailed characterization of the corrosion process under glycemic conditions.

IBM SPSS Statistics (Version 29.0; IBM Corp., Armonk, NY, USA) was used for data analysis.

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), Two-way ANOVA (material × glucose concentration), Repeated-measures ANOVA for immersion time, and Tukey's post hoc multiple comparison test. Statistical significance was established at $p < 0.05$.

Results

Surface Roughness

The results showed that there was a significant increase in surface roughness after immersion in the hyperglycemic artificial saliva. This increase depended on the concentration of glucose and length of the immersion period. Progressive increase in the roughness value was observed in S/S and NiTi archwire while minimal changes were observed in experimental period in β-Ti archwire.

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 significantly lower values (0.41 ± 0.03 µm) ($p < 0.001$).

Metal Ion Release

The released metallic ions concentration was found to be significantly higher with increasing glucose concentration and time of immersion. When hyperglycemia condition, Ni was most released from NiTi archwires. The release of Fe

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

and Cr, from the stainless steel, was also high in comparison to Ti release from beta-titanium, and Ti release from beta-titanium was relatively low throughout the study. Both two-way ANOVA (glucose concentration and immersion duration) showed significant effect on ion release ($p < 0.001$).

Colour changes were found in all the archwires after immersions. Color changes were measured of all archwires after immersions. 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 dipped in 30 mmol/L glucose. The color changes of beta-titanium were significantly more stable than with other materials.

SEM Evaluation

All the control specimens were found to be smooth and homogeneous by scanning electron microscopy.

The specimen was immersed in the hyper glycemic saliva, which then revealed a corrosion defect at the site, and it grew worse.

Minor irregularities and shallow pits were seen at glucose concentration 10 mmol/L.

At the higher glucose concentration (20 mmol/L), more severe corrosion was found: localized pitting and disruption of the passive oxide film were seen.

When the concentration is 30mmol/L glucose, significant corrosion could be 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 only isolated on the surface morphology of beta-titanium.

EDX Analysis

The chromium concentration decreased with increasing exposure time 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 percentage has risen, indicative of better oxide formation because of corrosion.

The elemental changes in beta-Titanium were very slight when compared with the other alloys.

Overall Corrosion Assessment

A RM-ANOVA showed differences in glucose concentration ($p < 0.001$), immersion duration ($p < 0.001$), and material type ($p < 0.001$).

Finally, there was a significant interaction between glucose concentration and immersion time ($p = 0.002$), indicating that the effect of glucose concentration on immersion time was greater with longer immersion times. The best material in terms of corrosion resistance was beta-titanium and the most corrosive was stainless steel with the greatest colour change. Nickel–titanium had the greatest nickel ion release, especially when exposed to the highest concentration of glucose for extended periods of time.

Discussion

The present study revealed that the high sugar content of artificial saliva had a significant effect on the corrosion rate of orthodontic archwires, and on the surface roughness, metal ion release and colour stability. The present observations indicate that the hypothesis that a correlation exists between hyperglycemia and alterations of the electrochemical properties of the oral cavity and hence the tendency to corrosion of metallic orthodontic materials is plausible. The present results indicated that hyperglycemic states may also be an important clinical factor that influences the longevity and biocompatibility of the orthodontic appliances, particularly in patients suffering from poorly controlled diabetes mellitus (DM), as the acidic pH, fluoride containing agents and mouthwashes are considered as corrosive factors [3-5]. All types of archwires have more roughness in both glucose concentration and immersion time, which was statistically significant. This finding is likely due to the gradual disturbance of the protective layer of passive oxide that develops naturally on metallic orthodontic alloys. Ss archwires had the most surface deterioration and iron and chromium ion release of the materials investigated. The main reason for the corrosion protection of stainless steel is the passive oxide film with a high chromium content, but the passive film can be destabilised by long exposure to an aggressive electrolyte and can increase electrochemical dissolution. Previously, 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 [6-8].

Nickel–titanium archwires had intermediate corrosion resistance with the highest levels of nickel ion release when compared to all other examined alloys. These observations have significant biological implications as nickel is one of the most common causes of hypersensitivity with orthodontic materials. The concentration of nickel released in this study was not beyond the limits of laboratory safety, but the cumulative release of nickel may be higher if the same concentration is present throughout the orthodontic treatment, particularly with unfavorable oral conditions. Like the above, 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 [4,8].

Beta-titanium archwires had the highest corrosion resistance and minimal surface degradation. The main reason for their superior performance is thought to be the fast 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 and less in SEM measurements than in S/S and NiTi archwires in every case.The lower surface deterioration and reduced Ti ion release were confirmed in all the SEM observations, EDX analysis and ICP-MS measurements compared with S/S and NiTi archwires. The same result has been observed in previous study that has tested titanium based orthodontic alloys under simulated oral conditions [4].

In the present study, the increasing amount of release of metallic ions with the passage of time also supports this idea that longer exposure of hyperglycemic saliva has an accelerating effect on electrochemical degradation of orthodontic alloys. Raised glucose levels can lead to elevated electrolyte conductivity and to oxidation–reduction reactions taking place at the metal surface, which can cause dissolution of nickel, chromium and iron. Moreover, the dose-response interaction between the glucose concentration and immersion duration suggests that hyperglycemic responses are additive and that the duration of exposure is important. These findings support 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 [4,6,7].

Conclusion

In this in-vitro study, within the limitations of the study, the corrosion related changes of orthodontic archwires were significantly accelerated by hyperglycemic artificial saliva. The surface roughness was increased, metal ion release increased, surface degradation increased, and the color stability of the sample was decreased with increasing glucose concentration in comparison with conventional artificial saliva. The more significant effects were observed with extended exposure, and therefore the concentration of the glucose solution and exposure time are both important in the corrosion process.

S. Archwires had the highest susceptibility to corrosion and released the highest amount of Fe and Cr ions among all evaluated materials. Nickel–titanium archwires exhibited moderate corrosion resistance and the highest amount of Ni ions released, while beta-Ti archwires had excellent corrosion resistance, a minimal amount of surface degradation, and the lowest

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

amount of metallic ion release. The results indicate that the chronic hyperglycemic state can have negative effects on the treatment results and survival of orthodontic appliances. Hence, careful material selection, frequent clinical management and good glycemic control should be considered in the orthodontic management of diabetic patients. In vivo studies are recommended in the future to confirm these results in real oral environment and to analyze how oral biofilm and mechanical loading of orthodontic materials interact in a hyperglycemic environment.

Study Limitations

In the interpretation of the results of the present study some limitations should be kept in mind.

This study was in vitro, and 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 like mastication, temperature changes or pH cycling or mechanical stresses was conducted.

The study was conducted on isolated archwire samples, and not 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 maximum immersion period of 28 days used. The longer the exposure time, the more representative of the duration of clinical orthodontic treatment.

The study only considered the corrosion behavior, the surface characteristics, colour stability and the release of metal ions, and nothing regarding changes to 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 studied in future.

To consolidate the findings of this study, further studies are required with the inclusion of long-term in vivo tests with patients suffering from diabetes, investigation of various orthodontic alloys and coatings, incorporation of oral biofilm models, and the combined effect of hyperglycemia, acidic liquids, fluoride exposure, and cyclic mechanical loading on orthodontic material degradation.

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Table 1. After 28 days, the surface roughness (Ra, µm) of orthodontic archwires was measured.

Archwire	Control	10 mmol/L	20 mmol/L	30 mmol/L	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

Table 2. The release of metals (µg/L) after 28 days.

Material	Ion	Control	10 mmol/L	20 mmol/L	30 mmol/L
Stainless Steel	Iron	1.84 ±0.21	2.71 ±0.26	3.94 ±0.35	5.83 ±0.42
Stainless Steel	Chromium	0.62 ±0.08	1.14 ±0.12	1.78 ±0.15	2.69 ±0.18
NiTi	Nickel	0.82 ±0.14	1.67 ±0.20	2.81 ±0.28	3.96 ±0.42
NiTi	Titanium	0.39 ±0.05	0.46 ±0.06	0.53 ±0.07	0.68 ±0.08
Beta-Titanium	Titanium	0.24 ±0.04	0.29 ±0.05	0.33 ±0.05	0.41 ±0.05

ANOVA: $p < 0.001$

Table 3. Color change (ΔE).

Archwire	Control	10 mmol/L	20 mmol/L	30 mmol/L
Stainless Steel	0.62 ±0.11	1.38 ±0.17	2.34 ±0.21	3.86 ±0.29
NiTi	0.59 ±0.1	1.26 ±0.16	2.07 ±0.18	3.21 ±0.24
Beta-Titanium	0.43 ±0.08	0.76 ±0.1	1.08 ±0.13	1.64 ±0.17

$p < 0.001$