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Research Article

Exploration of Metal Ion Release from Nickel-Chromium Denture Material: The Role of Saliva pH and Immersion Duration

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Abstract

Nickel chromium (NiCr) is a fixed denture material. Nickel has good physical, and mechanical properties, cheap, but it has low corrosion resistance. One way to increase corrosion resistance is to modify the metal surface by silver plating using the electroplating method. This study examines the effect of saliva pH and immersion time of NiCr metal with silver plating on the release of nickel, chromium, and silver ions. Laboratory experimental studies were 27 samples of NiCr with silver plating (Ø= 10 x 10 x 2 mm) immersed in saliva and divided into 9 groups (n=3): group I (pH 5 for 5 days), group II (pH 7 for 5 days), group III (pH 9 for 5 days), group IV (pH 5 for 10 days), group V (pH 7 for 10 days), group VI (pH 9 for 10 days), group VII (pH 5 for 15 days), group VIII (pH 7 for 15 days), group IX (pH 9 for 15 days). Nickel, chromium, and silver ions release was measured using atomic absorption spectrophotometry. The data obtained were analyzed using two-way ANOVA and post hoc LSD with a 95% confidence level (α =0.05). The results showed a significant difference between salivary pH and immersion time on the ion release ($p<0.05$). The release of nickel and silver ions increases at acidic pH, while the release of chromium ions increases at alkaline pH. Prolonged immersion (for 15 days) in saliva increases the release of nickel and chromium ions but decreases the release of silver ions.

Keywords

Nickel Chromium, Silver, Salivary pH, Immersion Time, Ions Release

1. Introduction

Fixed prosthodontic treatment aims to restore the function, aesthetics and comfort of the patient by replacing and restoring teeth with a non-removable denture. Fixed prosthodontic dental treatment encompasses the restoration of individual teeth with metal crowns, the replacement of one or more missing teeth with a fixed denture (GTC), and highly complex restorations supported by implants [1, 2]. Fixed dentures must exhibit compliance with several requirements, including those related to mechanics, physiology, hygiene, aesthetics, and phonetics [3]. All-metal fixed dentures are appropriate for single crown restorations on posterior teeth that require substantial strength and do not require aesthetic considerations. The advantages of metal crowns include enhanced retention and robust shape resistance, particularly in the context of short clinical crowns [4].

Nickel chromium (NiCr) is used as a metal material in the

fabrication of fixed dentures due to its favorable mechanical properties, including strength, hardness, and elastic modulus, as well as its relatively low-cost [5, 6]. The biocompatibility of nickel materials in the oral cavity remains a concern due to their relatively weak corrosion resistance, which results in the gradual degradation of the material through the release of metal ions into the body environment [7, 8].

Metal alloys in the oral cavity that interact with saliva will reach a state of thermodynamic equilibrium. Once equilibrium is reached, the alloy will either remain in its elemental form undergo oxidation to become ionic, or corrosion. The corrosion of metal alloys occurs when initially uncharged elements in the alloy lose electrons and become positively charged ions when released into solution [9]. Several factors, both internal and external, may cause corrosion. Internal factors that affect corrosion include metal composition and structure [9, 10]. External factors that can affect corrosion include the biological environment, metal processing methods, pH, and temperature [11-13].

The allergenic potential of silver is lower than that of other metals that have been used as coating materials, including titanium and chromium. The release potential of silver ions is relatively low and non-toxic [14, 15]. Silver is known to have good biocompatibility [16] and high antibacterial properties [17, 18]. Silver is less chemically reactive than other metals. A reduction in chemical reactivity may contribute to enhanced resistance to corrosion processes. Upon oxidation, a highly stable oxide layer is formed on the surface of the silver. The oxide layer acts as a protective barrier, preventing interaction between the metal surface with oxygen or corrosive substances. In their study, observed the release of silver ions from a titanium implant material with a silver nanoparticle coating when immersed in a phosphate-buffered saline (PBS) solution [14]. The release was observed to increase progressively over the 12-day observation period, reaching a concentration of 2.5 ppm by the end of the study. Following an additional 14-day period, the alteration in the release rate exhibited a saturated behavior, with the concentration of silver ions in the PBS solution approaching 2.5 ppm. It has been demonstrated that concentrations of silver ions in the human body exceeding 10 ppm are toxic [19].

The release of metal ions from prosthodontic materials, such as nickel-chromium alloys and silver coatings, can have a variety of negative impacts on human health [9]. Nickel and chromium ions can cause allergic reactions, dermatitis, and systemic toxicity if concentrations in the body exceed thresholds, with potential impacts on vital organs as well as cancer risk [18]. In addition, local tissue damage in the oral cavity, metabolic disorders, and negative effects on microbial flora can occur as a result of exposure to metal ions [16]. Although silver has antibacterial properties, high concentrations can affect the balance of the oral microbiota and cause harmful accumulation in the body. Therefore, close monitoring of ion release and management of prosthodontic materials is essential to minimize long-term health risks.

One method for the plating of silver on nickel-chromium alloy metals is electroplating. The electroplating method for metal plating offers the advantage of a solid coating with an even thickness distribution across the entire surface area of the workpiece [20]. One method of determining the corrosion resistance of nickel-chromium alloys with silver coatings is through the analysis of the concentration of released nickel, chromium, and silver ions from the alloy metal using atomic absorption spectrophotometry (AAS), coupled with the observation of the morphology and thickness of the silver layer on the nickel-chromium alloys [21].

The concentration of ions leached from NiCr alloy metal after it had been coated with titanium. The findings indicated that the application of titanium coatings effectively reduced the quantity of ions released from NiCr alloy metal [22]. Demonstrated that the application of a 5 μm thick silver coating to a cobalt chromium alloy metal (CoCr) resulted in a notable reduction in the release of cobalt and chromium metal ions in acidic saliva pH [23]. The present study makes a significant distinction from that of the previous researcher in terms of the material under examination. In particular, the present study focuses on a nickel-chromium alloy metal with a silver plating thickness of 5 μm [24, 25]. The aim of this study was to assess the impact of salivary pH and immersion time on the release of nickel, chromium, and silver ions from a denture material with silver plating.

2. Material and Methods

2.1. NiCr Sample Preparation

The NiCr sample was shaped into a square plate with dimensions of 10 x 10 x 2 mm using the NiCr metal casting method. Subsequently, the NiCr metal was polished with silicon carbide paper and sandblasted with aluminium dioxide. This process was employed to remove various types of dirt or other unwanted layers and to produce microporosity on the metal surface.

2.2. Silver Plating by Electrochemical Deposition

The application of a silver coating is achieved through the process of electrochemical deposition, which involves the use of an electrochemical reaction to facilitate the deposition of silver. The electrolyte solution, which serves as the source of silver ions, is carefully poured into the container. The sample is placed in the center of the anode and cathode. The source of silver ions, i.e. pure silver, is connected to the positive pole (anode), while the cathode is connected to the negative pole (anode). The cathode is composed of NiCr metal. The electrons emitted from the silver will migrate from the anode to the cathode, subsequently entering the electrolyte solution and bonding with the NiCr metal surface. Connect the positive cable (identified by a red coloration) to the anode and the

negative cable (identified by a black coloration) to the cathode. Subsequently, the black cable should be connected to the cathode, and the rectifier activated.

2.3. Ion Release Testing

Each sample was immersed in glass bottles with 25 ml of artificial saliva with pH 5, 7, 9, then marked according to the treatment group and stored in an incubator at 37 ℃. The pH of each group was monitored periodically using a pH meter. After 5, 10, and 15 days, the samples were removed from the glass bottles and the ion release in the soaking solution was quantified using an atomic absorption spectrophotometer (AAS). The data obtained are expressed as parts per million (ppm).

2.4. Research Subject

The samples used in this study were NiCr metal in the form of a square plate with a sample size of 10 mm x 10 mm x 2 mm that had been silver plated with a thickness of 5 μm. The sample size is determined using Federer's formula, the sample size is rounded to 3 per group, resulting in a total of 27 samples. The sample size is relatively small but is capable of producing high variability and wide confidence intervals, increasing the risk of type I (rejecting the correct zero hypothesis) and type II (failing to detect existing effects). Therefore, although the results of the study show an effect, they are carefully interpreted and confirmed with a larger sample size to ensure the validity and reliability of the findings.

1) Group I:

3 NiCr metal samples with 5 μm *silver* coating and immersed in pH 5 saliva for 5 days.

2) Group II:

3 NiCr metal samples with 5 μm *silver* coating and immersed in saliva pH 7 for 5 days.

3) Group III:

3 NiCr metal samples with 5 μm *silver* coating and immersed at saliva pH 9 for 5 days.

4) Group IV:

3 NiCr metal samples with 5 μm *silver* coating and immersed at saliva pH 5 for 10 days.

5) Group V:

3 NiCr metal samples with 5 μm *silver* coating and immersed at saliva pH 7 for 10 days.

6) Group VI:

3 NiCr metal samples with 5 μm *silver* coating and immersed at saliva pH 9 for 10 days.

7) Group VII:

3 NiCr metal samples with 5 μm *silver* coating and immersed at saliva pH 5 for 15 days.

8) Group VIII:

3 NiCr metal samples with 5 μm *silver* coating and immersed at saliva pH 7 for 15 days.

9) Group IX:

3 NiCr metal samples with 5 μm *silver* coating and immersed at saliva pH 9 for 15 days.

3. Results

As illustrated in Figure 1a, extensive corrosion gives rise to irregularly shaped holes or cracks in the oxide layer, thereby compromising its integrity. Figure 1b demonstrates the existence of smaller pits while the oxide layer remains intact. This indicates that the corrosion process is not particularly severe and that the oxide layer is still capable of protecting the metal surface. Figure 1c illustrates the effects of corrosion on multiple metal surfaces, accompanied by the formation of a thicker oxide layer.
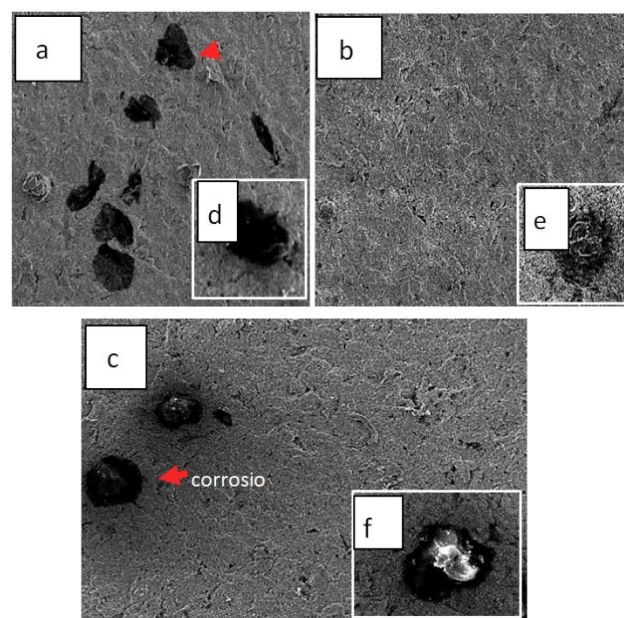

Figure 1. *Surface morphology of NiCr metal with silver plating under 500 x (a-c) and 1000 x (d-f) magnification: (a) pH 5 day 15; (b) pH 7 day 15; (c) pH 9 day 15.*

In Figure 1a, corresponding to pH 5 after 15 days of immersion, extensive corrosion is evident, as indicated by the formation of irregularly shaped holes and cracks in the oxide layer. The presence of these defects compromises the integrity of the protective oxide layer, allowing corrosive agents like chloride ions to penetrate deeper into the metal. This aggressive environment, characterized by a high concentration of H^+ ions, accelerates the breakdown of the oxide layer, leading to significant metal ion release. Observed damage reported that pitting corrosion caused by chloride was more severe under acidic conditions, where the oxide layer was more susceptible to dissolution.

Figure 1b, which shows the surface morphology at pH 7 after 15 days, reveals a less severe corrosion process. The

smaller pits observed suggest that while corrosion has initiated, the oxide layer remains largely intact, continuing to offer some level of protection to the metal surface. This indicates that at a neutral pH, the balance between oxidation and reduction reactions allows for the maintenance of a more stable oxide layer. The reduced rate of metal ion release under these conditions supports the idea that the neutral pH environment is less corrosive, allowing the oxide layer to effectively shield the underlying metal.

In contrast, Figure 1c, representing the metal surface at pH 9 after 15 days of immersion, shows a thicker and more uniform oxide layer despite the presence of some corrosion effects. The alkaline environment, with a lower concentration of H^+ ions and higher oxygen availability, facilitates the formation of a more robust oxide layer. This layer acts as a protective barrier, reducing the extent of corrosion and minimizing the release of metal ions. The formation of a thicker oxide layer at pH 9 is consistent with the protective behavior observed in alkaline environments, where oxygen plays a crucial role in stabilizing the oxide layer and preventing further degradation of the metal surface.
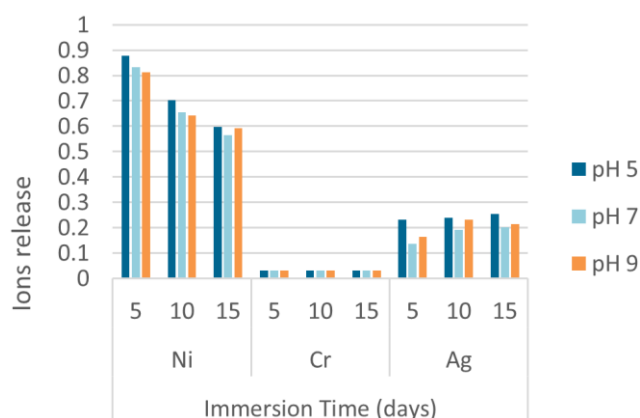

Figure 2. Comparison of Ni, Cr, and Ag ions released NiCr with silver plating.

Figure 2 shows that the release of nickel and chromium ions increased following an immersion period of five days, before subsequently decreasing on days ten and fifteen. Similarly, the release of silver ions increased following immersion periods of five, ten, and fifteen days. All data were normally distributed, thus allowing a two-way ANOVA parametric analysis to determine the significance of variables against the sample groups. The two-way ANOVA with 95% confidence on the interaction between the day variable (length of immersion) and the pH variable will result in significant differences in the release of nickel, chromium, and silver ions.

The corrosion resistance of NiCr metal with silver plating is contingent upon the formation of an oxide layer. The composition of the NiCr oxide layer is constituted of nickel oxide (NiO) and chromium oxide (Cr_2O_3). The ratio of NiO and Cr_2O_3 exerts a significant influence on the capacity of the oxide layer to interact with silver ions. The crystal structures of NiCr oxide layers differ, influencing the density, porosity and defects present in the oxide layer. A crystal structure with a higher porosity may facilitate the diffusion and penetration of silver ions into the oxide layer. This is in accordance with the diffusion theory, which posits that metal-metal adhesion will occur between the coating metal and the coated metal if there is particle exchange between the coating and the main metal, thus forming an atomic interdiffusion bond. Particle exchange occurs in the innermost layer of the coating metal and the outermost layer of the main metal.

The chemical bond between silver ions and the oxide layer (NiO and CrO_{23}) can influence the stability and durability of the interaction between silver ions and the oxide layer. The diffusion of silver ions into the oxide layer (NiO and CrO_{23}) will result in a reaction with oxygen, forming a silver oxide layer (Ag_2O). The interaction between Ag_2O and Cr_2O_3 results in the formation of an Ag-Cr-O complex compound. The interaction between Ag_2O and NiO layers will result in the formation of an Ag-Ni-O mixed layer. In addition to the aforementioned individual interactions, the Ag_2O, Cr_2O_3, and NiO layers will interact with each other as a double/mixed layer system. The composition of the mixed phase (Ag-Cr-O, Ag-Ni-O, and Ag-Ni-Cr-O) is contingent upon the interdiffusion rate of each ion and the conditions of layer formation, including temperature, ion pressure, and time. The crystal structure that is formed in the mixed phase will determine the adhesion properties between the layers. The formation of a robust adhesion between the coating and the substrate will prevent the coating from detaching, thereby enhancing the coating's resistance to corrosion. The interaction between the biological environment and the phase structure determines which elements will be released, the quantity released, and the duration of exposure [20].

Table 1. Shapiro-Wilk Normality Test.

Ion	Group	P-value Shapiro Wilk Test	P-value Levene test
Nickel	pH 5, H5	0,463	
	pH 7, H5	0,637	
	pH 9, H5	0,637	
	pH 5, H10	0,463	
	pH 7, H10	1,000	0,085
	pH 9, H10	0,637	
	pH 5, H15	1,000	
	pH 7, H15	1,000	
	pH 9, H15	1,000	
Chromium	pH 5, H5	0,843	0,080

Ion	Group	P-value	P-value
		Shapiro Wilk Test	Levene test
	pH 7, H5	0,780	
	pH 9, H5	0,878	
	pH 5, H10	0,945	
	pH 7, H10	0,114	
	pH 9, H10	0,616	
	pH 5, H15	0,878	
	pH 7, H15	0,637	
	pH 9, H15	0,780	
	pH 5, H5	0,637	
	pH 7, H5	0,637	
	pH 9, H5	1,000	
	pH 5, H10	1,000	
Silver	pH 7, H10	0,637	0,914
	pH 9, H10	1,000	
	pH 5, H15	1,000	
	pH 7, H15	1,000	
	pH 9, H15	1,000	

The mean nickel ion release exhibited the highest value following immersion in artificial saliva for a period of five days, subsequently demonstrating a decline on days 10 and 15. These findings demonstrate that the duration of immersion exerts a significant influence on the release of nickel ions. The initial highest mean result for nickel ion release is observed in Group I (pH 5, day 5). This phenomenon can be attributed to the thermodynamic instability of nickel ions in a salivary environment with an acidic pH. Consequently, the NiCr metal with silver plating undergoes oxidation into an ionic form. The $3d^8 4s^2$ electron configuration of nickel ions results in relatively weak Ni-O bonds in oxide compounds [26]. The duration of immersion influences the length of time the metal is in contact with the salivary fluid, thereby affecting the adhesion between the oxidation layers and increasing the release of Ni ions. Following a 15-day observation period, the highest nickel ion release was recorded at pH 5, reaching a concentration of 0.597 ppm. It can be reasonably assumed that the number of ions released per day is within the range of ±0.0398 ppm. The concentration of nickel ions released (0.0398 μg) is below the tolerable intake levels set by the American Dietary Guidelines (ADG) and the Canadian Environmental Protection Act (CEPA), which recommend a daily intake of 12 μg per kg of body weight. Additionally, the World Health Organization (WHO) has set a daily dose of 25-35 μg for nickel ions. The formation of the oxide layer results in an oxidation reaction on the surface of the nickel metal, which in-

hibits the diffusion of nickel ions into the surrounding environment. Consequently, the daily release of nickel ions will be lower over time in the presence of silver plating than in its absence. Excessive levels of nickel ions in saliva have the potential to cause adverse health effects for the user. In their study, Velasco-Ibáñez et al. (2020) [24] observed an increase in nickel ion levels in saliva and urine in patients using titanium and stainless steel nickel alloy devices in the oral cavity. The release of nickel ions from a range of metal alloys has been demonstrated to act as potent allergens, capable of eliciting cutaneous inflammation. These ions have the potential to penetrate the skin and activate epithelial cells, resulting in the production of various cytokines or chemokines [27].

Table 2. Results of two-way ANOVA test of nickel ion release.

Variables	F	P
pH	1352,925	0,000*
Day	38924,224	0,000*
Interaction of pH and Day	235,720	0,000*

F = calculated F value
p = probability or significance value
* = significant difference $p<0.05$

The results of the two-way ANOVA test, as presented in Table 2, underscore the significant influence of both pH and the duration of immersion on the release of nickel ions. The calculated F values for pH (1352.925) and immersion time (38924.224) both yield p-values of 0.000, indicating that these factors independently and in combination significantly affect the amount of nickel ions released ($p < 0.05$). The significant interaction between pH and the duration of immersion (F = 235.720, p = 0.000) suggests that the effect of pH on nickel ion release is not constant across different time points; rather, it varies depending on how long the metal has been immersed. This implies that the acidic environment and extended exposure time together accelerate the oxidation process, leading to higher nickel ion release, particularly within the first few days. As immersion time increases, the protective oxide layer forms more robustly, gradually decreasing nickel ion release. This dynamic interaction between pH and immersion duration is crucial for understanding the release kinetics of nickel ions in different conditions, highlighting the importance of managing both factors to minimize potential health risks.

The mean release of chromium ions (Table 2) demonstrated the highest value at pH 5 following immersion in artificial saliva for five days, subsequently exhibiting a decline on days 10 and 15. These findings demonstrate that the duration of immersion influences the release of chromium ions. Following a 15-day immersion period, the highest mean release of chromium ions was observed at pH 9 (0.0315 ppm), while the

lowest release was recorded at pH 7 (0.0308 ppm). The release of ions is a consequence of contact between the metal and the artificial salivary fluid. Artificial saliva is an electrolyte solution that can cause corrosion. The mean release of chromium ions across all groups fell below the intake standard chromium concentration. The nature of chromium renders it more resistant to corrosion than nickel. Furthermore, the chromium oxide layer formed is stronger, which results in a lower release of chromium ions. The chromium oxide layer is more compact and less permeable to water and oxygen, thereby impeding the diffusion of chromium ions into the surrounding environment.

Table 3. Post Hoc LSD test results of nickel ion release.

	pH 5, H5	pH 7, H5	pH 9, H5	pH 5, H10	pH 7, H10	pH 9, H10	pH 5, H15	pH 7, H15	pH 9, H15
pH 5, H5	-								
pH 7, H5	.043*	-							
pH 9, H5	.064*	.021*	-						
pH 5, H10	.174*	.130*	.109*	-					
pH 7, H10	.220*	.177*	.156*	.046*	-				
pH 9, H10	.235*	.191*	.170*	.061*	.014*	-			
pH 5, H15	.279*	.236*	.215*	.105*	.059*	.044*	-		
pH 7, H15	.312*	.269*	.248*	.138*	.092*	.077*	.033*	-	
pH 9, H15	.284*	.241*	.220*	.110*	.064*	.049*	.005*	-.028*	-

* shows significant difference $p<0.05$

H = day

The Post Hoc LSD test results presented in Table 3 further elucidate the significant differences in nickel ion release across various pH levels and immersion durations. The data reveals that the release of nickel ions is significantly different when comparing different pH levels at the same immersion time, as well as when comparing the same pH level across different days. For example, nickel ion release at pH 5 on day 5 is significantly different from that at pH 7 and pH 9 on the same day ($p < 0.05$). Similarly, there is a significant difference in nickel ion release when comparing pH 5 on day 5 to pH 5 on days 10 and 15. These findings suggest that both the acidic environment and prolonged exposure contribute to the increased release of nickel ions, with the most significant differences observed in the earlier stages of immersion. Moreover, as immersion time progresses, the differences in nickel ion release between the pH levels become more pronounced, particularly between the lower pH environments and the neutral or alkaline conditions. This indicates that the acidic environment continues to play a pivotal role in the release of nickel ions over time, likely due to the persistent influence of the acidic conditions on the breakdown of the protective oxide layer. The trend also suggests that as the immersion period extends to 15 days, the protective oxide layer's development and the metal's adaptation to the acidic environment may start to mitigate the release, albeit not completely, as indicated by the significant differences still present between the conditions.

As illustrated in Table 3, the mean values of silver ion release demonstrated an increase in concentration within each pH group on days 5, 10, and 15. This is a consequence of alterations to the standard electrode potential, which impact the electromotive force in the metal oxidation reaction. A reduction in standard electrode potential results in an increase in the electromotive force associated with the silver oxidation reaction. During the corrosion process, the oxidation of silver releases electrons more readily and rapidly when the standard electrode potential declines. The rate of acceleration or inhibition of the anodic process is contingent upon the rate of acceleration or inhibition of the cathodic process, or vice versa. The acceleration of the oxidation of silver results in an increase in the reduction reaction involving oxygen (O_2) in the corrosive environment, thereby accelerating the cathodic process in the corrosion reaction. The electrode potential is subject to alteration as a consequence of alterations in particle size. In their 2023 study, Ershov and Ershov observed that the fraction of dissolved metal and the rate of release of silver ions into solution increase with decreasing particle size. A reduction in particle size will result in an increase in electrode potential and an acceleration of the oxidation rate.

Table 4. Two-way ANOVA Test Results of Chromium Ion Release.

Variables	F count	P
pH	18,413	0,000*
Day	32,590	0,000*
Interaction of pH and Day	3,301	0,034*

Description Table 4:
F = calculated F value
p = probability or significance value
* = significant difference $p < 0.05$

Table 4 presents the results of the two-way ANOVA test for chromium ion release, revealing the significant effects of pH, immersion time, and their interaction on chromium ion release. The calculated F values for pH (18.413) and immersion time (32.590), both with p-values of 0.000, indicate that these factors significantly influence the release of chromium ions ($p < 0.05$). The interaction between pH and immersion duration, while significant (F = 3.301, p = 0.034), is less pronounced than in the case of nickel ion release. This suggests that while pH and immersion time independently affect the release of chromium ions, their combined effect is more subtle. Additionally, the significant interaction between pH and immersion time suggests that the effect of pH on chromium ion release changes over time, though this effect is not as dynamic as with nickel ions [28, 29]. Over extended immersion periods, it is likely that the release of chromium ions stabilizes as the metal surface becomes increasingly passivated by the oxide layer. This passivation may reduce the rate at which chromium ions are released, especially in less acidic or neutral environments [30].

Table 5. Post Hoc LSD test results of chromium ion.

	pH 5, H5	pH 7, H5	pH 9, H5	pH 5, H10	pH 7, H10	pH 9, H10	pH 5, H15	pH 7, H15	pH 9, H15
pH 5, H5	-								
pH 7, H5	.00017	-							
pH 9, H5	.00008	-.00009	-						
pH 5, H10	.00042*	.00024*	.00034*	-					
pH 7, H10	.00062*	.00045*	.00054*	.00020	-				
pH 9, H10	.00021	.00003	.00013	-.00020	-.00041*	-			
pH 5, H15	.00053*	.00035*	.00045*	.00011	-.00009	.00031*	-		
pH 7, H15	.00098*	.00081*	.00090*	.00056*	.00036*	.00077*	.00045*	-	
pH 9, H15	.00032*	.00015	.00024*	-.00009	-.00030*	.00011	-.00020	-.00066*	-

* shows significant difference $p < 0.05$
H = day

The Post Hoc LSD test results in Table 5 reveal significant differences in chromium ion release across various pH levels and immersion durations. The data indicates that at pH 5 on day 5, chromium ion release is significantly different from that at pH 7 and pH 9 on the same day, with similar significant differences observed across subsequent days ($p < 0.05$). These differences highlight the strong influence of the acidic environment (pH 5) on chromium ion release, particularly in the earlier stages of immersion. As immersion time progresses to 10 and 15 days, the differences in ion release between pH levels remain significant, particularly between acidic (pH 5) and neutral or alkaline conditions (pH 7 and pH 9). This suggests that while the acidic environment continues to promote chromium ion release over time, the overall effect diminishes as the immersion period extends, likely due to the stabilization of the protective oxide layer on the metal surface.

The consistent significance of these differences underscores the importance of pH in influencing chromium ion release and reinforces the need to monitor and control pH levels in environments where metal exposure is prolonged, such as in medical and dental applications, to mitigate potential health risks.

Table 6. Two-way ANOVA test results of silver ion release.

Variables	F count	p
pH	6714,077	0,000*
Day	4163,385	0,000*

Variables	F count	p
Interaction of pH and Day	652,885	0,000*

F = calculated F value

p = probability or significance value

* = significant difference p<0.05

Table 6 presents the two-way ANOVA test results for silver ion release, demonstrating a highly significant influence of both pH and immersion time on the release of silver ions. The extremely high F values for pH (6714.077) and immersion time (4163.385), with p-values of 0.000, indicate that these factors play a dominant role in determining the amount of silver ions released (p < 0.05). Additionally, the significant interaction between pH and day (F = 652.885, p = 0.000) suggests that the effect of pH on silver ion release varies significantly over time. This strong interaction implies that the acidic environment not only accelerates the release of silver ions but also that this effect is particularly pronounced in the initial days of immersion. As time progresses, the relationship between pH and silver ion release becomes more complex, possibly due to the formation of a protective layer on the metal surface that influences the release kinetics. These findings emphasize the importance of carefully controlling both pH and exposure duration to manage silver ion release effectively, especially in environments where prolonged exposure to metals is expected, such as in medical devices or dental materials, to prevent excessive ion release and associated health risks.

Table 7. Post hoc LSD test results of silver ion release.

	pH 5, H5	pH 7, H5	pH 9, H5	pH 5, H10	pH 7, H10	pH 9, H10	pH 5, H15	pH 7, H15	pH 9, H15
pH 5, H5	-								
pH 7, H5	.096*	-							
pH 9, H5	.069*	-.026*	-						
pH 5, H10	-.005*	-0.101*	-.075*	-					
pH 7, H10	.041*	-.055*	-.028*	.046*	-				
pH 9, H10	.001	-.094*	-.068*	.007*	-.039*	-			
pH 5, H15	-.022*	-.118*	-.092*	-.017*	-.063*	-.024*	-		
pH 7, H15	.030*	-.065*	-.039*	-.036*	-.010*	.029*	.053*	-	
pH 9, H15	.018*	-.077*	-.051*	.024	-.022*	.017*	.041*	-.012*	-

* shows significant difference p<0.05

H = day

The results of the two-way ANOVA test indicate that there is an effect of salivary pH and immersion time on the release of nickel and chromium ions from NiCr metal with silver plating (Tables 5 and 7). The observed results are attributable to differences in salivary pH. Upon encountering H ions, nickel metal ions will engage in oxidation and reduction (redox) reactions. In the chemical reaction, the $Ni2^+$ ions will lose two electrons, while the H+ ions will receive two electrons, resulting in the production of hydrogen gas. The loss of two electrons results in the oxidation of the nickel metal ion ($Ni2^+$) to nickel metal. Conversely, hydrogen ions accept both electrons and undergo reduction to form hydrogen gas (H_2). The oxide layer on the nickel metal surface acts as a barrier, preventing excessive reactions between nickel metal and H^+ ions. The longer the alloy metal is immersed in saliva, the greater the interaction of metal ions with H^+ ions and the dissolution of the oxide layer.

Saliva with a pH of 5 has a higher concentration of H^+ ions and a lower concentration of oxygen ions than pH 7. It can be observed that as the pH of saliva decreases, the oxide layer formed on the metal surface becomes less stable and more susceptible to dissolution. In their research, pH 2.5 the highest passivation current density was recorded [31]. This indicates that the passive film is not an adherent, robust, and compact layer on the metal surface. Chloride is a highly corrosive and aggressive agent. The presence of chloride ions can result in the deterioration of the oxide layer on the metal surface, thereby increasing its exposure and vulnerability to corrosive attack. The presence of chloride ions can facilitate electrochemical reactions that accelerate corrosion, such as metal oxidation and oxygen reduction. The greater the acidity of the electrolyte, the more probable it is that the concentration of metal ions will be released into the electrolyte solution [32].

The results of the LSD post hoc test of chromium ions

demonstrate a significant difference in pH between the saliva samples (pH 5, 7, 9) after 15 days of immersion, while no significant difference was observed during the immersion period on days 5 and 10. These results may be attributed to the inherent stability and corrosion resistance of chromium metal in a range of environments, including those with acidic or alkaline pH levels, such as saliva. The chromium can form protective surface oxides due to oxygen activity on the metal surface, which then forms chromium oxide (CrO_2) [33]. This oxide layer can protect the underlying metal from corrosion. The longer the NiCr metal is immersed in saliva with a pH of 5 and 9, the more the oxide layer is damaged, leading to corrosion of the metal. This phenomenon can be attributed to the reaction between chromium and hydrogen ions present in saliva, which ultimately leads to the formation of water-soluble chromium oxide compounds. The presence of elevated concentrations of hydrogen ions will result in a loss of stability, thereby facilitating electrochemical reactions. Saliva with an acidic pH will result in an increase in cathode reaction, which will subsequently lead to corrosion. The oxide layer can be dissolved when it binds to chloride ions in saliva, resulting in pitting corrosion and the penetration of anionics such as chloride into the electrolyte solution. Subsequently, the corrosion process continues with the propagation of pits and the breakdown of the oxide layer. Pitting corrosion is the consequence of electrolyte attack (chloride ions) and the corrosion process commences at the site of damage to the oxide layer [34]. The oxide layer is particularly challenging to re-form when damaged in a solution containing chloride ions [35].

The concentration of silver ions released will gradually decline until it reaches a saturation point, which occurs when the oxide layer is fully formed and has reached a sufficient thickness. At the point of saturation, the rate of silver ion release becomes markedly slow due to the formation of an oxide layer that effectively prevents or inhibits direct contact between the metal and the surrounding environment. Ag ions were released at a high rate during the initial two days, with a concentration of 0.25-0.28 ppm maintained thereafter [36]. The silver ions are a cooperative oxidation process that requires protons and dissolved O_2 [37]. Protonation has the effect of weakening and breaking Ag-O bonds, which results in a greater release of silver ions at acidic pH than at neutral pH [38-40].

The surface morphology of the NiCr metal with silver plating was observed using a scanning electron microscope (SEM) to ascertain the effects of immersion in saliva with a pH of 5 for 15 days (group 7). The results demonstrated the formation of irregular holes or cracks, indicative of local corrosion. This is attributable to the presence of chloride ions, which have the potential to impede the formation of an oxide layer on the metal surface, thereby increasing the likelihood of crevice corrosion. The silver oxide layer (Ag_2O) is capable of reacting with chloride ions present in saliva, resulting in the formation of AgCl (silver chloride), which is inherently unstable. The release of silver ions can damage the surface in-

tegrity of the silver oxide layer, resulting in the formation of pitting. This allows the electrolyte solution (saliva) to reach the oxide layer below, accelerating the corrosion process and the release of nickel and chromium ions. The chloride ions present in saliva solutions are responsible for pitting corrosion. The increase in chloride ions in saliva with a pH of 5 results in a decrease in the concentration of dissolved oxygen, thereby inhibiting the formation of the oxide layer and accelerating the rate of crevice corrosion [41, 42].

On day 15 (group 8), observations of the NiCr metal surface immersed in saliva with a pH of 7 revealed a corrosion pattern characterized by the presence of smaller cracks and holes, along with a discernible oxide layer. This is due to the occurrence of thermodynamic equilibrium between the components of H^+ and oxygen ions in saliva, which interact with ions on the surface of metal alloys through redox reactions. Thermodynamic equilibrium between the oxidation and reduction of metal ions in saliva can be achieved when the reaction speed of these processes reaches a similar level.

The observation of NiCr metal surfaces immersed in saliva with a pH of 9 on the 15th day (Group 9) revealed corrosion effects on some metal surfaces accompanied by the formation of a thicker oxide layer. This is due to the fact that alkaline saliva (pH 9) has a lower concentration of H+ ions and a higher concentration of oxygen ions than pH 7. The oxide layer is formed as a result of the exposure of ions on the metal surface to oxygen. The oxide layer serves as a protective barrier against further corrosion of the metal surface. The interaction of silver ions with oxygen results in the formation of an oxide layer, which prevents full oxidation and ensures the stability of protective properties, thereby preventing the release of nickel and chromium ions.

The present study demonstrated that the pH of saliva and the immersion time of NiCr alloy metal as an adhesive denture material exerted a significant influence on the release of NiCr metal ions with silver plating. Following a 15-day immersion period, there was an increase in the release of silver ions and a concomitant decrease in the release of nickel ions. The release of ions from the NiCr metal fixed denture with silver plating can be attributed to several factors, including contact with salivary electrolytes (chloride ions, dissolved oxygen, salivary pH, proteins, and amino acids), temperature, porosity, and instability of the coating, and the intrinsic properties of NiCr metal. Accordingly, further analysis of the morphological observation results using scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDX) is required to identify the specific ion composition of the NiCr metal surface with a silver coating.

4. Discussion

The results of this study indicate a clear influence of salivary pH and immersion time on the release of nickel, chromium, and silver ions from NiCr alloy with silver plating. The two-way ANOVA results confirm that variations in salivary pH and

extended immersion times significantly affect the release of metal ions. This can be attributed to the different electrochemical reactions that occur at varying pH levels. Specifically, a lower pH increases the concentration of H^+ ions, which facilitates redox reactions leading to the dissolution of the protective oxide layer on the metal surface [22]. This finding, suggested that the degradation of the oxide layer in acidic environments leads to increased nickel ion release [43].

Chromium, known for its corrosion-resistant properties, exhibited significant differences in ion release at different pH levels, particularly after 15 days of immersion. The inherent ability of chromium to form a stable oxide layer under varying pH conditions explains why significant differences were observed only at extended immersion times. This behavior is consistent with Bardal (2004), who noted that chromium's ability to form protective oxides under different environmental conditions enhances its resistance to corrosion [24, 25]. However, prolonged exposure to acidic and alkaline environments (pH 5 and 9) eventually leads to the breakdown of this protective layer, resulting in increased corrosion and ion release.

The study also highlights the complex behavior of silver ions during immersion. The increase in silver ion release over time can be linked to changes in the standard electrode potential, which accelerates the anodic process and consequently the cathodic reduction of oxygen. Smaller particle sizes of silver contribute to a higher rate of ion release due to increased surface area and altered electrode potential [44]. However, the formation of a protective oxide layer over time eventually slows the release of silver ions, indicating a dynamic balance between oxidation and the formation of a passivation layer [45].

Surface morphology analysis using SEM revealed corrosion patterns consistent with the observed ion release trends. In acidic conditions (pH 5), the presence of chloride ions exacerbates pitting corrosion, damaging the oxide layer and facilitating the release of nickel and chromium ions [46]. The study confirms Renita et al.'s (2016) findings that chloride ions play a significant role in pitting corrosion, especially in environments with low pH. In contrast, the formation of a thicker oxide layer at alkaline pH (pH 9) serves as a protective barrier, reducing the release of nickel and chromium ions. This suggests that the stability of the oxide layer is heavily dependent on both pH and the presence of aggressive ions like chloride in the saliva.

5. Conclusions

It has been observed that there is a correlation between the pH of saliva and the release of ions from silver-plated NiCr metal. The release of ions from silver-plated NiCr metal has been shown to be influenced by the pH of saliva. Specifically, acidic saliva has been demonstrated to increase the release of nickel and silver ions, while alkaline saliva has been observed to enhance the release of chromium ions. A 15-day immersion of the NiCr metal with silver plating in saliva was found to result in an increased release of nickel and chromium ions,

while the release of silver ions was observed to decrease over the same period.

For future research, it is recommended to conduct a more in-depth study on the effects of a wider range of salivary pH levels, including extreme conditions below pH 5 and above pH 9, to understand the limits of corrosion resistance in silver-plated NiCr metal under various corrosive environments. Additionally, further research could involve surface morphology analysis using techniques such as X-ray diffraction (XRD) and Fourier-transform infrared spectroscopy (FTIR) to study the chemical changes in the protective oxide layer. Other studies may focus on varying the thickness of the silver coating and exploring alternative coating materials, such as gold or platinum, to evaluate their effectiveness in minimizing ion release. Moreover, it is crucial to assess the biocompatibility and toxicity of nickel, chromium, and silver ion release, particularly in long-term medical applications.

Abbreviations

NiCr	Nickel-Chromium
NiO	Nickel Oxide
Cr_2O_3	Chromium Oxide

Acknowledgments

Author Contributions

Putri Dhianita Pratiwi: Conceptualization and design of the study, data collection, data analysis, and drafting of the initial manuscript.

Murti Indrastuti: Methodology development, laboratory supervision, and technical guidance during experiments.

Titik Ismiyati: Statistical analysis, data interpretation, and critical revision of the intellectual content of the manuscript.

Conflicts of Interest

The authors declare no conflicts of interest.

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Biography

Putri Dhianita Pratiwi is a lecturer at the University of Muhammadiyah Semarang, Department of Prosthodontics. Completed a bachelor's degree in dentistry and a dentist profession at Airlangga University, and is currently pursuing a Prosthodontics specialist education program at Gadjah Mada University, Yogyakarta Indonesia. He is interested in research in the field of removable dentures.

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Titik Ismiyati is a Lecture in the Faculty of Dentistry, Universitas Gadjah Mada Indonesia, my research has been focusing on material for prosthodontic. I also study the effect of chitosan mixing with basic materials dental acrylic resin to growth Candida albicans, transversal strength, tensile strength and hardness researched about mechanical and physical properties, residual monomers, and anti inflammation on the denture. In parallel of teaching and research, I also involve in managing a dental and oral hospital as director of medical and education services and teaching for undergraduate and graduate program of Faculty of Dentistry.

Research Field

Putri Dhianita Pratiwi: Dental Materials Science
Murti Indrastuti: Prosthodontics, Material Science, Biomaterials in Dentistry
Titik Ismiyati: Biomaterial, Mechanical strengh, Remodelingcluster - Prosthodontics