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Effect of Immersion in 0.12% Chlorhexidine Solution on Corrosion and Unloading Force of Rhodium-Coated NiTi Archwires

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ABSTRACT

The types of orthodontic archwires commonly used in orthodontic treatment are NiTi non-coated and rhodium-coated. Corrosivity and unloading force on the wires may undergo changes due to the use of mouthwash, specifically chlorhexidine 0.12%. The aim of this study was to investigate the effect of immersing NiTi non-coated and rhodium-coated with a diameter of 0.014" in chlorhexidine 0.12% solution on their corrosivity and unloading force after immersion for 1, 7, and 14 days. A total of 48 NiTi non-coated and rhodium-coated archwires were divided into 12 groups (n = 4 each) cut to a length of 30 mm. They were grouped based on immersion media (chlorhexidine 0.12% and artificial saliva) and immersion time (1, 7, 14 days). In the chlorhexidine immersion group, the wires were immersed and shaken for one minute twice a day, then rinsed and immersed in artificial saliva. Samples were tested using atomic absorption spectrophotometry (AAS) and a three-point bending test. Statistical analysis was performed using two-way ANOVA, DMRT 5%, and linear regression. The NiTi non-coated archwires immersed in chlorhexidine exhibited high corrosivity of 0.0083 ppm and unloading force of 2.0275 N after 14 days. SEM analysis of NiTi non-coated archwires revealed a rougher surface with larger porosities, while rhodium-coated archwires showed peeling of the coating and pitting. The immersion of non-coated NiTi archwire and rhodium-coated archwire in chlorhexidine solution on the 14th day has the highest corrosivity and unloading force compared to immersion durations on days 1 and 7.

Keywords: *Chlorhexidine; corrosivity; NiTi archwire; rhodium-coated archwire; unloading force*

INTRODUCTION

Rinsing with mouthwash can eliminate interdental bacteria that are inaccessible by toothbrushes. Mouthwash is used to reduce plaque accumulation during active orthodontic treatment (Rakhman, 2020). Rinsing before treatment procedures with mouthwash containing 0.12% to 10.2% can reduce the microbial count in saliva (Nik *et al.*, 2013). Mouthwash containing 0.12% chlorhexidine can reduce contamination during prophylaxis for orthodontic patients undergoing fixed treatment, effectively reducing plaque accumulation, gingival inflammation, and gingival bleeding (Santos *et al.*, 2014).

Orthodontic wires were exposed to saliva and mouthwash, releasing ions. One metal ion with a high tendency to be released was nickel ion (Schmaltz & Arenholt, 2009).

Archwire immersion is the process of soaking archwires in a solution. Archwire immersion is done continuously for 1, 7, and 14 days. On the first day, the solution is collected after archwire immersion. This is done to analyse the release of nickel ions on the first day of immersion, and the same process is repeated on days 7 and 14. The release of nickel ions from the archwire in 0.12% chlorhexidine solution for 1, 7, and 14 days begins to be detected on the 1st day of immersion, with the highest release occurring on the 14th day (Leliana, 2010).

Corrosion is the deterioration of metal material due to electrochemical reactions with the environment, involving the dissolution of elements on the metal surface, known as ion release (Leliana, 2010). The release of nickel ions can cause contact dermatitis, carcinogenic, cytotoxic, and genotoxic effects because NiTi archwires contain a significant amount of nickel (approximately 48% to 55%) (Barcelos *et al*., 2013). Along with ion release during the corrosion process, NiTi archwire can experience a decrease in stiffness, leading to suboptimal tooth movement (Nik *et al*., 2013). Corrosivity can be tested with atomic absorption spectrophotometry (AAS) which is a technique for measuring the amount of chemical elements in a sample by measuring the radiation absorbed by a specific chemical element (Kristianingsih *et al*., 2014).

The initial phase is characterised by rapid tooth movement within 24 to 48 hours of applying orthodontic force (Ariffin *et al*., 2011). Optimal tooth movement in the initial phase requires continuous yet gentle distribution of orthodontic force. The magnitude of orthodontic force generated falls within the biological limit accepted by the periodontal tissues, ranging from 0.50 to 0.70 N. This force should not exceed 2.00 to 3.00 N. The NiTi archwires are selected due to their properties of superelasticity and shape memory (Aghili *et al*., 2015).

Unloading force is influenced by several factors, including the wire material, wire section (diameter), and wire length (Lindauer *et al*., 2015). The release of nickel ions can affect the mechanical properties of the wire, one of which is the unloading force. The wire will lose elasticity and become stiffer, because the nickel ions in the wire contribute to enhancing its flexibility (Carpenter, 2014). The superelastic properties of NiTi archwires can be determined through the load-deflection process, where a highly elastic wire is subjected to force, causing deflection. When the load is removed, the wire can return to its original shape, transmitting force distributed to the dentoalveolar area and causing tooth movement (Santoro *et al*., 2001).

As time progresses, there is an increasing focus on aesthetics, leading to the emergence of fixed orthodontic components that match the colour of teeth. One such component is the NiTi aesthetic archwire with an additional coating. This archwire is divided into two types: composite archwires and coated metallic archwires. Composite archwires are made of glass fibre, while coated metallic archwires are made of metal and coated with tooth-coloured polymer. Tooth-coloured polymers can include epoxy, Teflon, silver (Ag), polymer, and rhodium (Haryani & Ranabhatt, 2016). The precious metal rhodium has characteristics such as being white in colour, more aesthetic than other metals, chemically stable, and ha excellent resistance to wear (Kim *et al*., 2014). Therefore, the present study aimed to investigate the influence of immersing in a 0.12% chlorhexidine solution for 1, 7, and 14 days on the corrosivity and unloading force of non-coated NiTi archwires and rhodium-coated NiTi archwires used in the initial phase of orthodontic treatment.

MATERIAL AND METHODS

The research conducted in this study follows a laboratory experimental approach. The research design and materials underwent review and approval by the ethics committee of the Faculty of Dentistry-Prof. Soedomo Dental Hospital, Universitas Gadjah Mada, Indonesia (Ref. No.: 176/UNI/KEP/FKG-RSGM/EC/2023).

The wires used in this study are non-coated NiTi archwires and rhodium-coated NiTi archwires with a preformed round shape, 0.014 inches in size, from the brand Tomy (Japan). The wires were measured using callipers, and then marked along 30 mm at both ends to obtain a straight portion. Subsequently, the wires were cut using cutting pliers and randomly divided according to the number of groups. The treatment groups were as follows: Group 1 (4 non-coated NiTi archwires immersed in artificial saliva for 1 day); Group 2 (4 non-coated NiTi archwires immersed in 0.12% artificial saliva for 7 days); Group 3 (4 non-coated NiTi archwires immersed in 0.12% artificial saliva for 14 days); Group 4 (4 rhodium-coated NiTi archwires immersed in artificial saliva for 1 day); Group 5 (4 rhodium-coated NiTi archwires immersed in artificial saliva for 7 days); Group 6 (4 rhodium-coated NiTi archwires immersed in artificial saliva for 14 days); Group 7 (4 non-coated NiTi archwires immersed in 0.12% chlorhexidine solution for 1 day); Group 8 (4 non-coated NiTi archwires immersed in 0.12% chlorhexidine solution for 7 days); Group 9 (4 non-coated NiTi archwires immersed in 0.12% chlorhexidine solution for 14 days); Group 10 (4 rhodium-coated NiTi archwires immersed in 0.12% chlorhexidine solution for 1 day); Group 11 (4 rhodium-coated NiTi archwires immersed in 0.12% chlorhexidine solution for 7 days); Group 12 (4 rhodium-coated NiTi archwires immersed in 0.12% chlorhexidine solution for 14 days). A 0.12% chlorhexidine solution was placed in polypropylene tubes, 20 ml each.

The first step was to conduct a test of the elemental content and surface structure of the rhodium-coated NiTi archwire using a scanning electron microscope (SEM) (JEOL JSM IT-200, Japan). The archwires were simulated in the oral environment. All samples were immersed in 20 ml of artificial saliva with a pH of 6.8 during the observation period (1, 7, 14 days), and the 0.12% chlorhexidine solution was simulated twice a day for 1 minute. Samples immersed in the 0.12% chlorhexidine solution were shaken manually for 1 minute to simulate gargling, then rinsed with flowing water, for 5 seconds and subsequently immersed in artificial saliva.

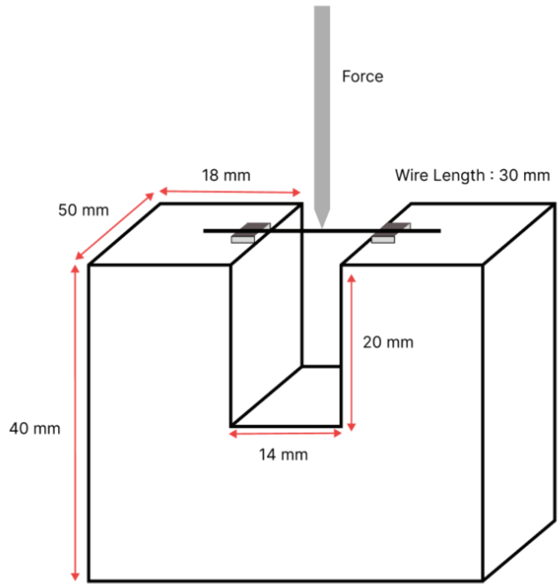

Fig. 1 Illustration of the unloading force test.

Corrosivity measurement was performed using the AAS test which is conducted by immersing the sample solution into a syringe, typically around 1 ml to 2 ml, then subjecting it to combustion to produce atomic vapour. The atomic vapour is irradiated by a cathode lamp with a wavelength of 232 nm to determine the nickel ion concentration.

Unloading force measurement was conducted using the three-point bending test on a universal testing machine. A jig consisting of a U-shaped aluminium plate was mounted on the universal testing machine at a 90° angle to the floor and fixed to the non-moving testing machine component (Fig. 1). Two standard metal brackets were fixed to the jig with a distance of 14 mm. The archwire was placed in the bracket slots so that when the testing machine was run, the moving part would press the wire (loading process) and return to the initial position (unloading process), allowing the unloading force to be measured. The unloading force measurement was performed by operating the universal testing machine, pressing the archwire downward until it deflected by 3 mm (loading process). When reaching a 3 mm deflection, the testing apparatus moved back up (unloading process), and at a deflection of 1.5 mm, the magnitude of the unloading force was recorded (Iflah *et al.*, 2017).

RESULTS

The results of nickel ion release and unloading force for 12 groups are displayed in Tables 1 and 2.

Table 1 Mean and SD of nickel ion release (ppm) in 0.014″ non-coated NiTi and rhodium-coated NiTi archwires in 0.12% chlorhexidine solution and artificial saliva for 1, 7, and 14 days

Immersion time	Goup	Mean (ppm) ± SD
1 day	NiTi - Saliva	0.0033 ± 0.0011
	Rhodium - Saliva	0.0024 ± 0.0010
	NiTi - CHX	0.0038 ± 0.0010
	Rhodium - CHX	0.0037 ± 0.0009
7 days	NiTi - Saliva	0.0065 ± 0.0007
	Rhodium - Saliva	0.0058 ± 0.0006
	NiTi - CHX	0.0074 ± 0.0005
	Rhodium - CHX	0.0069 ± 0.0004
14 days	NiTi - Saliva	0.0072 ± 0.0007
	Rhodium - Saliva	0.0064 ± 0.0007
	NiTi - CHX	0.0083 ± 0.0004
	Rhodium - CHX	0.0077 ± 0.0004

Table 2 Mean and SD of unloading force (newton) in 0.014″ non-coated NiTi and rhodium-coated NiTi archwires in 0.12% chlorhexidine solution and artificial saliva for 1, 7, and 14 days

Immersion time	Group	Mean (newton) ± SD
1 day	NiTi - Saliva	0.4025 ± 0.1484
	Rhodium - Saliva	0.3162 ± 0.0998
	NiTi - CHX	0.6350 ± 0.115
	Rhodium - CHX	0.4600 ± 0.1327
7 days	NiTi - Saliva	0.5212 ± 0.1505
	Rhodium - Saliva	0.5025 ± 0.1172
	NiTi - CHX	0.7987 ± 0.2699
	Rhodium - CHX	0.6900 ± 0.2443
14 days	NiTi - Saliva	0.9562 ± 0.1366
	Rhodium - Saliva	0.9525 ± 0.1362
	NiTi - CHX	2.0275 ± 0.2618
	Rhodium - CHX	1.5525 ± 0.4212

Table 1 shows that the highest mean nickel ion release is in the group of non-coated NiTi archwires immersed in chlorhexidine solution for 14 days, while the lowest nickel ion release is in the group of rhodium-coated NiTi archwires immersed in artificial saliva for 1 day. Table 2 indicates that the highest mean unloading force is in the group of samples immersed in 0.12% chlorhexidine solution for 14 days, suggesting the smallest unloading force, while the largest unloading force is in the group of samples immersed in artificial saliva for 1 day.

The results of the two-way ANOVA test indicate that the release of nickel ions and the unloading force produced by rhodium-coated NiTi archwire and non-coated NiTi archwire have a probability value of $p < 0.05$. This suggests a significant difference in the corrosivity and unloading force generated by rhodium-coated NiTi archwire and non-coated NiTi archwire over the immersion periods of 1, 7, and 14 days (Table 3 and Table 4). The two-way ANOVA results were followed by the Duncan multiple range test (DMRT) at a 5% significance level to determine distinct groups.

Table 3 Results of two-way ANOVA for nickel ion release in 0.014″ non-coated NiTi and rhodium-coated NiTi archwires in 0.12% chlorhexidine solution and artificial saliva for 1, 7, and 14 days

Source of variation	Db	F	*p*
Immersion time	2	124,132	0.000*
Immersion media and wire type	3	7,314	0.001*
Interaction between immersion time and media/wire type	6	1,131	0.003*

Note: * *p* < 0.05

Table 4 Results of two-way ANOVA for unloading force in 0.014″ non-coated NiTi and rhodium-coated NiTi archwires in 0.12% chlorhexidine solution and artificial saliva for 1, 7, and 14 days

Source of variation	Db	F	*p*
Immersion time	2	78,664	0.000*
Immersion media and wire type	3	17,153	0.000*
Interaction between immersion time and media/wire type	6	4,169	0.003*

Note: * *p* < 0.05

Based on the DMRT test in Table 5, it is shown that the highest nickel ion release is observed in NiTi non-coated archwire immersed in 0.12% chlorhexidine solution for 14 days (notation "a"). This is followed by NiTi non-coated archwire immersed in saliva and rhodium-coated archwire immersed in 0.12% chlorhexidine for 14 days (notation "ab"), as well as NiTi non-coated archwire immersed in 0.12% chlorhexidine solution for 7 days (notation "ab"), with no significant difference in nickel ion release. Next is the combination of treatments, NiTi rhodium-coated archwire immersed in chlorhexidine and artificial saliva for 7 days (notation "bc"), rhodium-coated archwire immersed in saliva for 14 days (notation "bc"), and NiTi non-coated archwire immersed in saliva for 7 days (notation "c"), which significantly differs from other treatments. The treatment combination indicating the lowest corrosivity test is NiTi rhodium-coated archwire immersed in saliva for 1 day (notation "e"). This is not significantly different from NiTi non-coated archwire and NiTi rhodium-coated archwire immersed in saliva for 1 day (notation "de"), as well as NiTi non-coated archwire immersed in saliva for 1 day (notation "d").

Table 5 DMRT at 5% significance level for nickel ion release in 0.014" non-coated NiTi and rhodium-coated NiTi archwires in 0.12% chlorhexidine solution and artificial saliva for 1, 7, and 14 days

Treatment combination	Mean	Sig.
H1- NiTi Saliva	0.0033	de
H1 - Rhodium Saliva	0.0024	e
H1 - NiTi CHX	0.0038	d
H1 - Rhodium CHX	0.0037	d
H7- NiTi Saliva	0.0058	c
H7 - Rhodium Saliva	0.0065	bc
H7 - NiTi CHX	0.0074	ab
H7 - Rhodium CHX	0.0069	bc
H14- NiTi Saliva	0.0072	ab
H14 - Rhodium Saliva	0.0064	bc
H14 - NiTi CHX	0.0083	a
H14 - Rhodium CHX	0.0077	ab

Notes: Sig = Significanc; H1 = Immersion on day 1; H7 = Immersion on day 7; H14 = Immersion on day 14; UJD = DMRT; a, b, c, d, e, f = Notations in the DMRT 5% test. Numbers followed by the same letter in the same column do not differ significantly based on the DMRT 5%.

Results from the DMRT 5% in Table 6 indicate that high unloading values are shown by the interaction of NiTi non-coated archwires immersed in clorhexidine for 14 days (notation "a"), significantly differing from other treatment interactions. Following that is the combination of NiTi rhodium-coated archwires immersed in chlorhexidine for 14 days (notation "b"), significantly differing from other treatments. The subsequent treatments showing similar unloading results are NiTi non-coated archwires (notation "c") and NiTi rhodium-coated archwires (notation "c") immersed in artificial saliva for 14 days, NiTi non-coated archwires (notation "cd") and NiTi rhodium-coated archwires (notation "cde") immersed in chlorhexidine for 7 days, and NiTi non-coated archwires immersed in chlorhexidine for 1 day (notation "cdef"). The combination of treatment indicating the lowest unloading is NiTi rhodium-coated archwires immersed in saliva for 1 day (notation "f"), not significantly different from NiTi non-coated and NiTi rhodium-coated archwires immersed in saliva for 7 days (notation "def"), NiTi rhodium-coated archwires immersed in chlorhexidine for 1 day (notation "def"), and NiTi non-coated archwires immersed in artificial saliva for 1 day (notation "ef").

The surface characteristics of the wires are examined using a scanning electron microscope (SEM). Samples for SEM testing are randomly selected from NiTi non-coated and NiTi rhodium-coated archwire groups before treatment, as well as groups with the highest ion release for NiTi non-coated and NiTi rhodium-coated archwires after 14 days of immersion. The magnification used is 200 times. Darker colour compared to the wire's surface represents porosity and surface damage on the wire.

In Figs. 2a and 3a, the surfaces of the NiTi non-coated and NiTi rhodium-coated archwires before treatment appear smooth and free from scratches. In Fig. 2b and 2c, after immersion for 14 days, the NiTi non-coated archwire displays a rougher surface with fine lines and increased porosity. Fig. 3b and 3c show that the NiTi rhodium-coated archwire, immersed for 14 days, exhibits surface changes, peeling of the coating layer, and the presence of small pits (pitting corrosion).

Table 6 DMRT 5% for unloading force on NiTi non-coated and NiTi rhodium-coated archwires with a diameter of 0.014" in 0.12% chlorhexidine solution and artificial saliva for 1, 7, and 14 days

Treatment combination	Mean	Sig.
H1- NiTi Saliva	0.4025	ef
H1 - Rhodium Saliva	0.3162	f
H1 - NiTi CHX	0.635	cdef
H1 - Rhodium CHX	0.46	def
H7- NiTi Saliva	0.5212	def
H7 - Rhodium Saliva	0.5025	def
H7 - NiTi CHX	0.7987	cd
H7 - Rhodium CHX	0.69	cde
H14- NiTi Saliva	0.9525	c
H14 - Rhodium Saliva	0.9562	c
H14 - NiTi CHX	2.0275	a
H14 - Rhodium CHX	1.5525	b

Notes: Sig = Significanc; H1 = Immersion on day 1; H7 = Immersion on day 7; H14 = Immersion on day 14; UJD = DMRT; a, b, c, d, e, f = Notations in the DMRT 5% test. Numbers followed by the same letter in the same column do not differ significantly based on the DMRT 5%.

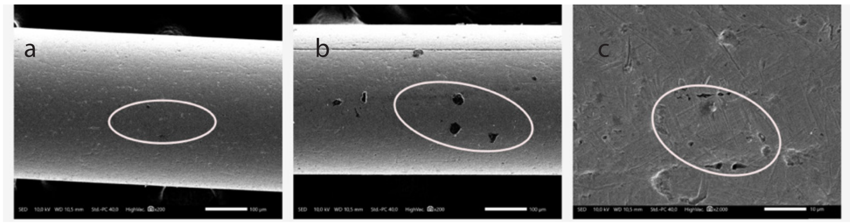

Fig. 2 (a) Morphological characteristics of NiTi non-coated archwire: untreated; (b) immersed for 14 days, 200x magnification; (c) Immersed for 14 days, 2,000x magnification. The circles indicate areas of corrosion and surface damage.

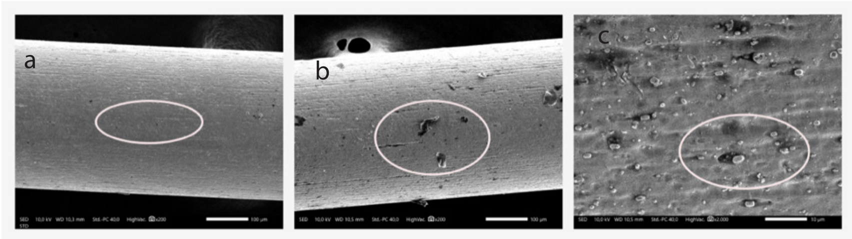

Fig. 3 (a) Morphological characteristics of NiTi rhodium-coated archwire: untreated; (b) immersed for 14 days, 200x magnification; (c) immersed for 14 days, 2,000x magnification. The circles indicate areas of corrosion and surface damage.

DISCUSSION

Nickel ion release during immersion in a 0.12% chlorhexidine solution is higher than in the artificial saliva. This is because the acid present in chlorhexidine mouthwash contains high H^+ particles that increase when reacting with metal, accelerating the corrosion rate (Ahmad *et al.*, 2014). Among the groups studied, the NiTi non-coated archwire group showed the highest average release of nickel ions, measuring 0.0083 ppm after 14 days of immersion in a 0.12% chlorhexidine solution. This is because the surface of the NiTi non-coated archwire is directly exposed to the environment, enhancing reactions with chemicals like chlorhexidine mouthwash that can cause corrosion. The NiTi rhodium-coated archwire group, with 1 day of immersion in artificial saliva, has the smallest average, 0.0024 ppm. This is because the rhodium layer on the coated wire serves as a protective layer, reducing direct exposure of the base metal to elements causing corrosion. Rhodium is also known as a corrosion-resistant metal. A longer immersion period (14 days) provides more time for chemical reactions between the material and the solution, resulting in a greater release of nickel ions. In the present study, the release of nickel ions on the first day was 0.0024 ppm, and on the 14 days, it was 0.0083 ppm. Therefore, in the present study, the nickel concentration did not cause any symptoms of toxicity (toxic nickel dose is 2.5 mg/ml; lethal oral nickel dose is 50 mg/kg body weight) (Luft *et al.*, 2009). The detected nickel levels are lower than toxicity levels, and they are also below the daily intake limit (200 µg). WHO states that an intake of 0.2 ppm nickel/kg can cause systemic manifestations (Luft *et al.*, 2009). A study by Karnam *et al.* (2012) on immersion of NiTi and stainless steel archwires in artificial saliva for 12 weeks (90 days) resulted in a total ion release less than the cumulative daily intake. However, these levels are sufficient to induce allergic reactions.

The pH of 0.12% chlorhexidine solution, which is 5.5. The chloride ions (Cl-) in the solution can increase the corrosion rate on the archwire's surface. The higher the concentration of chloride ions in the solution, the stronger the oxidation potential, and corrosion will occur more rapidly (Brar *et al.*, 2015). This is evident in the SEM examination (Fig. 2b and 2c) where the NiTi non-coated archwire immersed in a 0.12% chlorhexidine solution for 14 days appears rougher due to damage to its surface and has larger porosities due to the chemical reaction with the corrosive agent. SEM testing of NiTi rhodium-coated archwires in chlorhexidine immersion for 14 days (Fig. 3b and 3c) proves that the coating layer begins to peel, indicating degradation on the wire's surface, with evidence of pitting showing small points eroded from the rhodium layer.

Corrosion can release nickel ions because the NH element in the chemical structure of chlorhexidine will bond with nickel, forming NH−Ni–NH bonds (Ahmad *et al.*, 2014). This bond is a coordinative covalent bond that is more stable than an ionic bond, leading to a greater release of nickel ions in chlorhexidine solution compared to artificial saliva. The contents of artificial saliva can induce corrosion processes by damaging the oxide layer on the archwire surface, resulting in the release of metal ions, including nickel ions. Binding occurs when phosphate binds to nickel, forming nickel phosphate ($Ni_3(PO_4)^2$), which can attract nickel ions on the wire. Additionally, nickel ions are soluble in saliva, and the duration of wire contact with saliva can affect the release of nickel ions (Tang *et al.*, 2015).

The test results in Table 2 show that the NiTi non-coated archwire group immersed in a 0.12% chlorhexidine solution for 14 days has the highest average load, 2.0275 N. This is because the chlorhexidine solution during the extended immersion period can trigger corrosive reactions or structural changes in the NiTi non-coated archwire, as evidenced by the highest nickel ion release in that sample group. This can affect the mechanical properties of the wire, including elastic modulus and material strength, which, in turn, can influence unloading force (Fernandes *et al.*, 2011). NiTi rhodium-coated archwire group, immersed in artificial saliva for 1 day, exhibited the lowest average load, measuring 0.3162 N. The rhodium coating provides extra protection to the archwire against corrosion or changes in mechanical properties and unloading force (Cho *et al.*, 2002). Changes in unloading force are influenced by the extent of nickel ion release that occurs.

The released nickel ions can affect the mechanical properties of the wire, including unloading force. The archwire will lose its elasticity and become stiffer because the nickel ions in the wire play a role in increasing the flexibility of the wire. The archwire can produce effective unloading forces to facilitate tooth movement. Unloading force is an essential factor that orthodontists pay attention to during orthodontic treatment, as this force has a direct impact on tooth movement (Carpenter, 2014). Optimal orthodontic force produces efficient tooth movement without damaging tooth structure or periodontal tissues. Determining the exact figure for the ideal force becomes challenging because it depends on various factors, including tooth size and the type of movement needed. The desired orthodontic force is in the range of 0.15 to 5 N (Parvizi & Rock, 2003). In this study, the results of unloading force measurements on NiTi non-coated and NiTi rhodium-coated archwires (Table 2) are still within the optimal force range for tooth movement (Parvizi & Rock, 2003).

CONCLUSION

In conclusion, the 0.12% chlorhexidine solution causes corrosivity and an increase in unloading force in both NiTi non-coated and NiTi rhodium-coated archwires, NiTi non-coated archwires exhibit higher levels of corrosivity and unloading force compared to NiTi rhodium-coated archwires, and immersion of NiTi non-coated and NiTi rhodium-coated archwires in a 0.12%

chlorhexidine solution on day 14 results in the highest levels of corrosivity and unloading force compared to immersion durations on day 1 and day 7.

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