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Original Article

FERRUM ION RELEASE AND STIFFNESS CHANGES OF STAINLESS STEEL ORTHODONTIC WIRE IMMERSED IN DIFFERENT CONCENTRATIONS OF *PIPER BETLE LINN* MOUTHWASH

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ABSTRACT

Objective: Successful orthodontic treatment is related to patient oral hygiene. Using mouthwash is one way to maintain good oral hygiene. Orthodontic archwire that placed in oral cavity has contact with all components in mouth, including chemical mouthwash that using by the patient. This leading to a corrosion process that would affected the mechanical properties of the arch wire. Herbal mouthwash become an alternative to maintain patient's oral hygiene. This study was to examined ferrum ion release and stiffness value of stainless steel orthodontic wire immerse in artificial saliva, 0.2% chlorhexidine, and 1%, 2%, and 3% *Piper betle Linn* mouthwash.

Methods: Seventy-five stainless steel orthodontic wires were immersed in artificial saliva, 0.2% chlorhexidine, and 1%, 2%, 3% *Piper betle Linn* mouthwash. Each wire was inserted into a polypropylene tube containing 10 ml solution then placed in an incubator at 37 °C for 1,7 and 14 d. The solution was used to measure ferrum ion release by ICP-OES, and the wire was dried to test the stiffness values by a Universal Testing Machine.

Results: There was a significant increase in ferrum ion release and a significant decrease in stiffness in all immersion groups. The results has significant differences between groups ($p<0.05$).

Conclusion: The lowest ferrum ion release and the highest stiffness value between *Piper betle Linn* mouthwash were found in 3% *Piper betle Linn* mouthwash. It because there is more tannin content in 3% *Piper betle Linn* mouthwash than 1% and 2% *Piper betle Linn* mouthwash.

Keywords: Stainless steel wire, *Piper betle Linn* mouthwash, Ferrum ion release, Corrosion, Stiffness

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INTRODUCTION

Orthodontic wire is one of the active components that affect the success of orthodontic treatment [1]. Stainless steel orthodontic wire is commonly used because it has excellent mechanical properties, such as high resilience, high stiffness, ease of shape, corrosion resistance, and good biocompatibility. The composition of stainless steel wire is 71% iron (Fe), which is the main content, 18% chromium (Cr), 8% nickel (Ni), and 0.2% carbon (C) [2, 3].

Successful orthodontic treatment is closely related to good oral hygiene. Patients undergoing orthodontic treatment is needed to maintain good oral hygiene by teeth brushing and using mouthwash [4]. Chlorhexidine has been considered the gold standard for oral hygiene. Chlorhexidine has been proven to have broad-spectrum antibacterial properties, so it can prevent plaque accumulation by reducing the number of *Streptococcus mutans*. However, several studies report that chlorhexidine mouthwash can cause the release of metal ions [4, 5].

Ion release dramatically affects the characteristics of the wire, especially the stiffness, because stiffness is needed in tooth movement, especially bodily movement. The stiffness property is inversely proportional to flexibility; if stiffness is reduced, the wire will be more flexible, leading to decreased wire performance and disturb the important function of the wire to lead tooth movement [6]. The release of ferrum ions can affect the strength of orthodontic wire, starting with the formation of micro pit corrosion that can affect the sliding mechanism and biocompatibility of orthodontic treatment. Micro pit corrosion can increase the friction of the orthodontic wire, affecting tooth movement, the moment ratio of force to the dentition, and the center rotation of the teeth, leading to loss of anchorage and prolonging the duration of orthodontic treatment [7].

Piper betle Linn is one of Indonesian herbal plant that consists of flavonoids, tannins, alkaloids, and saponins as an antibacterial, anti-inflammatory, and antioxidant properties. *Piper betle Linn* can be used as mouthwash, an alternative to chemical mouthwash. The minimum inhibitory concentration (MIC) of *Piper betle Linn* mouthwash to inhibit the growth of Streptococcus mutans is 1% [8, 9].

Study on the ion release and mechanical properties of stainless steel wire has been carried out, but no study have been performed the effect of *Piper betle Linn* mouthwash on stainless steel orthodontic wire's physical and mechanical properties such as ferrum ion release and stiffness. This study aimed to determine the ferrum ion release and stiffness value of stainless steel orthodontic wires after immersion in artificial saliva, 0.2% chlorhexidine, and 1%, 2%, and 3% *Piper betle Linn* mouthwash.

MATERIALS AND METHODS

This study research used an experimental laboratory research with a post-test only control group design. The ethical clearance from the Komisi Etik Penelitian Kesehatan (KEPK) at the Faculty of Medicine Universitas Sumatera Utara was attained upon commencement of the study (Reference No: 1125/KEPK/USU/2023) on 14th November 2023.

Samples

Seventy-five 0.016-inch stainless steel orthodontic wires (American Orthodontics, USA) with 6 cm length were used as samples. The samples were divided into fifteen groups of five for each group. Amount of sample was calculated according to Federer's formula which equal for five samples for each group.

Samples are new stainless steel wire from sealed packaging with ISO (International Organization for Standardization) standards. Artificial saliva, 0.2% chlorhexidine mouthwash (Minosep®), and 1%,2%, and 3% *Piper betle Linn* mouthwash were used as liquid solutions. Artifisial saliva were made of composition: 9.3 g/l $Na_2HPO_4 12H_2O$, 0.47 g/l NaCl, 0.57 g/l KCl, 0.04 g/l CaCl, 0.06 g/l MgCl. These samples are divided into groups: Group 1A: Stainless steel wire immersed in artificial saliva and stored in an incubator at 37 °C for 1 day. Group 2A: Stainless steel wire immersed in 0.2% chlorhexidine mouthwash and stored in an incubator at 37 °C for 1 day. Group 3A, 4A, 5A: Stainless steel wire immersed in 1%, 2%, 3% *Piper betle Linn* mouthwash, and incubated at 37C for 1 day. Group 1B: Stainless steel wire immersed in artificial saliva and incubated at 37 °C for 7 d. Group 2B: Stainless steel wires immersed in 0.2% chlorhexidine and incubated at 37 °C for 7 d. Group 3B, 4B, 5B: Stainless steel wire immersed in 1%, 2%, 3% *Piper betle Linn* mouthwash, and incubated at 37 °C for 7 d. Group 1C: Stainless steel wire immersed in artificial saliva and incubated at 37 °C for 14 d. Group 2C: Stainless steel wire immersed in 0.2% chlorhexidine and incubated at 37 °C for 14 d. Group 3C, 4C, 5C: Stainless steel wire immersed in 1%, 2%, 3% *Piper betle Linn* mouthwash and incubated at 37 °C for 14 d.

The produce of *Piper betle Linn* mouthwash

Before produce *Piper betle Linn* extract, we identify betle leaves on Medanese herbarium plants systematics laboratory Universitas Sumatera Utara. Then, a total of 500 g fresh *Piper betle Linn* leaves were washed and then dried at 50 °C drying cabinet for three days. Then, *Piper betle Linn* leaves were blended until there were 30 g of simplisia. Simplisia was immersed using 300 ml of 96% ethanol for maceration 1 and 150 ml of 96% ethanol for maceration 2. Then, the entire macerate was evaporated using a vacuum rotavapor tool at 40 °C, then produced 6 g *Piper betle Linn* extract. This extract then put into phytochemical screening to determine secondary metabolite.

Piper betle Linn extract, weighed according to the concentration of mouthwash, into a mortar. Add CMC-Na and mixed until homogeneous. Then, add sorbitol, methylparaben, and peppermint oil. Finally, add distilled water up to 100 ml (table 1).

Table 1: Formulation of *Piper betle Linn* mouthwash

Ingredients	Concentration			Function
	1%	2%	3%	
Extract of *Piper betle Linn* leaves	1 g	2 g	3 g	Active ingredients
Sodium Carboxymethyl Cellulose (CMC Na)	8 g	8 g	8 g	*Suspending agent*
Sorbitol	10 ml	10 ml	10 ml	Sweetener
Methylparaben	0.05 g	0.05 g	0.05 g	Preservatives
Peppermint oil	q. s.	q. s.	q. s.	Fragrance
Distilled water	ad 100 ml	ad 100 ml	ad 100 ml	Solvent

The procedure of wire immersion

Before the samples were immersed, the pH meter measured the pH level of each immersion solution (*Inolab ph 7310*). The results showed: artificial saliva (pH 7), 0.2% chlorhexidine (pH 5.9), and *Piper betle Linn* mouthwash in 1% (pH 6.5), 2% (pH 6.8), and 3% (pH 7) concentration. Each sample was put into a polypropylene tube with close lid consisting of one wire and then put in an incubator (*Memmert*) at 37°C. Polypropylene tube sealed with close lid to prevent solution evaporation while incubating in the incubator that would affect solution concentration and acidity (pH level). Ferrum ion release from the solution measured by Inductively Coupled Plasma Optical Emission Spectrometry/ICP-OES (*Thermo Scientific)*, then the wire is dried and surface morphology was analyzed by Scanning Electron Microscope/SEM (*Hitachi, TM-3000*). Wire were tested for stiffness values using a Universal Testing Machine/UTM (*Tensilon RTF-1350*).

The procedure of the ferrum ion release test and surface morphology analysis

After incubation, each solution was measured to determine the amount ferrum ion release using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). To measure ferrum ion release, 2 ml of liquid sample put into a 50 ml volumetric flask. Then, 48 ml of concentrated HNO_3 as acidic matrix was added for liquid sample preparation. The solution was transferred into a beaker. Then, 6 ml of solution was pipetted into a tube. Commercial standard metal solution is used for calibration. Intensity of the standard metal solution was measured to prepare a calibration curve. Samples were tested using ICP-OES with a wavelength of 248,3 nm (fig. 1). The surface structure of the wire after immersion was analyzed using Scanning Electron Microscope (SEM). One sample was taken from each group to evaluated.

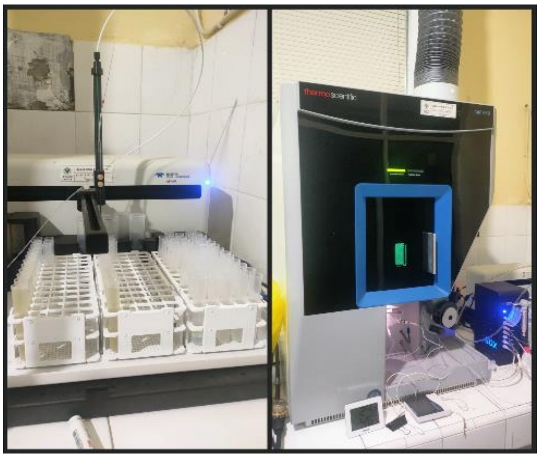

Fig. 1: Inductively coupled plasma optical emission spectrometry (ICP OES)

The procedure of the wire stiffness test

The procedur of the wire stiffness test using three-point bending test method. After the wire was dried, each wire was put into a Universal Testing Machine (UTM) then operated computerized with a crosshead speed of 0.5 mm/second. A 6 cm wire sample placed into the test equipment. The provisions of 1 cm on the right and left of the wire to hold wire and 4 cm wire will be in the middle. The wire in the middle will be subjected to a load with a diameter of 10 mm right in the center of the wire. The load will be dropped on the wire with a depth of 4 mm. The stiffness test is performed until the wire deforms (fig. 2).

Fig. 2: Universal testing machine (UTM)

Statistical analysis

The results obtained are processed statistically with a computerized program and using Statistical Package for the Social Sciences (SPSS) software for statistical analysis.

RESULTS

Identification of *Piper betle Linn* leaves

The result of the identification of *Piper betle Linn* leaves showed the following taxonomy.

Kingdom: Plantae

Division: Spermatophyta

Class: Dicotyledoneae

Ordo: Piperales

Familia: Piperaceae

Genus: Piper

Species: *Piper betle* L.

Phytochemical screening

Phytochemical screening of *Piper betle Linn* extract showed the existence of flavonoid, glycoside, saponin, tannin, and triperten/steroid (table 2).

Table 2: Result of phytochemical screening

Secondary metabolite	Indicators	Result
Alkaloid	Dragendroff	-
	Bouchardat	-
	Meyer	-
Flavonoid	Magnesium powder+Amil Alcohol+HCl_p	+
Glycoside	Molish+H_2SO_4	+
Saponin	Hot water/shaken	+
Tannin	$FeCl_3$	+
Triperten/Steroid	Lieberman-Bourchat	+

Ferrum ion release

Mean value of ferrum ion release after immersion for 1, 7, and 14 d increased with the increasing of immersion time. The highest Ferrum ion release was found in 0.2% chlorhexidine, followed by 1% *Piper betle Linn* mouthwash, 2% *Piper betle Linn* mouthwash, *and* 3% *Piper betle Linn* mouthwash, and the lowest was in artificial saliva (fig. 3).

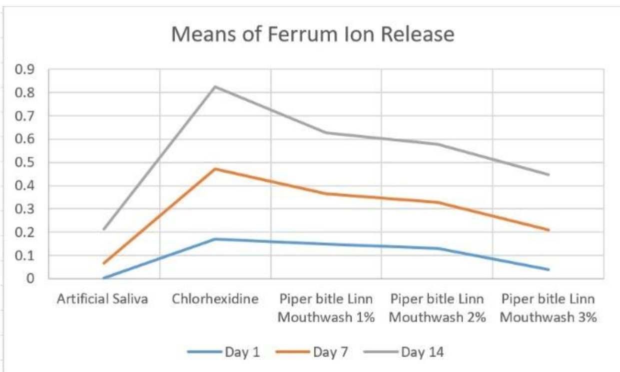

Fig. 3: Mean value of ferrum ion release after immersion for 1, 7, and 14 d

Two-way ANOVA analyzed ferrum ion release of stainless steel orthodontic wires at immersion solution showed significant difference at p=0.001 (p<0.05), at immersion time showed significant difference at p=0.001 (p<0.05), and between immersion solution and immersion time showed significant difference at p=0.001 (p<0.05) (table 3). Post hoc tukey test result between immersion groups showed signicant difference of ferrum ion release except between 1% and 2% *Piper betle Linn* mouthwash (table 4). Post hoc tukey test result between immersion time showed significant difference in all time groups at p=0.001 (p<0.05) (table 5).

Table 3: Two-way ANOVA test in ferrum Ion release based on time and solution of immersion

Variables	Ferrum ion release	
	F	p-value
Immersion Solution	310.179	0.001*
Immersion Time	528.795	0.001*
Immersion Solution* Immersion Time	9.571	0.001*

*p<0.05

Table 4: Post hoc tukey test result of ferrum ion release between immersion solution groups

Immersion solution		Mean difference (I-J)	Std. Error	Sig. (p-value)
(I)	(J)			
Artifisial Saliva	0.2 % Chlorhexidine	-.20393*	.006071	<.001
	1 % *Piper betle Linn* mouthwash	-.13833*	.006071	<.001
	2 % *Piper betle Linn* mouthwash	-.12200*	.006071	<.001
	3 % *Piper betle Linn* mouthwash	-.07827*	.006071	<.001
0.2 % Chlorhexidine	Artifisial Saliva	.20393*	.006071	<.001
	1 % *Piper betle Linn* mouthwash	.06560*	.006071	<.001
	2 % *Piper betle Linn* mouthwash	.08193*	.006071	<.001
	3 % *Piper betle Linn* mouthwash	.12567*	.006071	<.001
1 % *Piper betle Linn* mouthwash	Artifisial Saliva	.13833*	.006071	<.001
	0.2 % Chlorhexidine	-.06560*	.006071	<.001
	2 % *Piper betle Linn* mouthwash	.01633	.006071	.067
	3 % *Piper betle Linn* mouthwash	.06007*	.006071	<.001
2 % *Piper betle Linn* mouthwash	Artifisial Saliva	.12200*	.006071	<.001
	0.2 % Chlorhexidine	-.08193*	.006071	<.001
	1 % *Piper betle Linn* mouthwash	-.01633	.006071	.067
	3 % *Piper betle Linn* mouthwash	.04373*	.006071	<.001
3 % *Piper betle Linn* mouthwash	Artifisial Saliva	.07827*	.006071	<.001
	0.2 % Chlorhexidine	-.12567*	.006071	<.001
	1 % *Piper betle Linn* mouthwash	-.06007*	.006071	<.001
	2 % *Piper betle Linn* mouthwash	-.04373*	.006071	<.001

*p<0.05

Table 5: Post hoc tukey test result of ferrum ion release between immersion time groups

Immersion time		Mean difference (I-J)	Std. Error	Sig. (p-value)
(I)	(J)			
1 d	7 d	-.09120*	.004703	<.001
	14 d	-.15192*	.004703	<.001
7 d	1 d	.09120*	.004703	<.001
	14 d	-.06072*	.004703	<.001
14 d	1 d	.15192*	.004703	<.001
	7 d	.06072*	.004703	<.001

*p<0.05

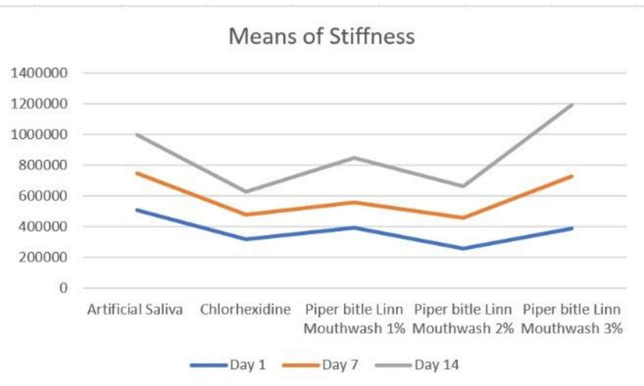

Fig. 4: Means of stiffness on stainless steel orthodontic wire after immersion for 1, 7, and 14 d

Wire stiffness

Mean value of stainless steel orthodontic wire stiffness after immersion in 1, 7, and 14 d decreased over time. The highest stiffness value was found in artificial saliva solution, followed by *Piper betle Linn* mouthwash at 3%, 2%, and 1%, and the lowest stiffness value was found in 0.2% chlorhexidine mouthwash (fig. 4).

Two-way ANOVA analyzed stiffness values of stainless steel orthodontic wires at immersion solution showed significant differences p=0.001 (p<0.05), at immersion time showed significant difference p=0.001 (p<0.05), and between immersion solution and immersion time showed significant difference p=0.001 (p<0.05) (table 6). Post Hoc Tukey Test result between immersion groups showed significant difference of stiffness value p=0.001 (p<0.05) (table 7). Post Hoc Tukey Test result between immersion time showed significant difference in all time groups at p=0.001 (p<0.05) (table 8).

pH Value

pH value before immersion showed the lowest value in 0.2% chlorhexidine mouthwash, followed with 3% *Piper betle Linn* mouthwash, 2% *Piper betle Linn* mouthwash, 1% *Piper betle Linn* mouthwash, and artifisial saliva. pH value after immersion decreased with increasing time in all immersion groups (table 9).

Table 6: Two-way ANOVA test in stiffness value based on time and solution immersion

Variables	Stiffness	
	F	p-value
Immersion solution	1910.882	0.001*
Immersion time	1333.957	0.001*
Immersion solution* immersion time	9.680	0.001*

*p<0.05

Table 7: Post hoc tukey test result of stiffness between immersion solution groups

Immersion solution		Mean difference (I-J)	Std. error	Sig. (p-value)
(I)	(J)			
Artifisial Saliva	0.2 % Chlorhexidine	8839668.84	112682.683	<.001
	1 % *Piper betle Linn* mouthwash	6678751.23	112682.683	<.001
	2 % *Piper betle Linn* mouthwash	4369458.41	112682.683	<.001
	3 % *Piper betle Linn* mouthwash	2333148.41	112682.683	<.001
0.2 % Chlorhexidine	Artifisial Saliva	-8839668.8	112682.683	<.001
	1 % *Piper betle Linn* mouthwash	-2160917.5	112682.683	<.001
	2 % *Piper betle Linn* mouthwash	-4470210.4	112682.683	<.001
	3 % *Piper betle Linn* mouthwash	-6506520.7	112682.683	<.001
1 % *Piper betle Linn* mouthwash	Artifisial Saliva	-6678751.2	112682.683	<.001
	0.2 % Chlorhexidine	2160917.61	112682.683	<.001
	2 % *Piper betle Linn* mouthwash	-2309292.8	112682.683	<.001
	3 % *Piper betle Linn* mouthwash	-4345603.1	112682.683	<.001
2 % *Piper betle Linn* mouthwash	Artifisial Saliva	-4369458.4	112682.683	<.001
	0.2 % Chlorhexidine	4470210.43	112682.683	<.001
	1 % *Piper betle Linn* mouthwash	2309292.82	112682.683	<.001
	3 % *Piper betle Linn* mouthwash	-2036310.3	112682.683	<.001
3 % *Piper betle Linn* mouthwash	Artifisial Saliva	-2333148.1	112682.683	<.001
	0.2 % Chlorhexidine	6506520.71	112682.683	<.001
	1 % *Piper betle Linn* mouthwash	4345603.10	112682.683	<.001
	2 % *Piper betle Linn* mouthwash	2036310.28	112682.683	<.001

*p<0.05

Table 8: Post hoc tukey test result of stiffness between immersion time groups

Immersion time		Mean difference (I-J)	Std. Error	Sig. (p-value)
(I)	(J)			
1 d	7 d	1752818.10000*	87283.630747	<.001
	14 d	4473589.71600*	87283.630747	<.001
7 d	1 d	-1752818.10000*	87283.630747	<.001
	14 d	2720771.61600*	87283.630747	<.001
14 d	1 d	-4473589.71600*	87283.630747	<.001
	7 d	-2720771.61600*	87283.630747	<.001

*p<0.05

Table 9: pH value before and after immersion

Immersion solutions	pH before immersion	pH after immersion		
		1 D	7 D	14 D
Artifisial Saliva	7	6.9±0.10	6.7±0.10	6.5±0.16
0.2% Chlorhexidine	5.9	5.7±0.14	5.6±0.08	5.3±0.16
1% *Piper betle Linn* mouthwash	6.5	6.4±0.10	6.1±0.16	5.9±0.07
2% *Piper betle Linn* mouthwash	6.8	6.7±0.10	6.5±0.07	6.3±0.16
3% *Piper betle Linn* mouthwash	7	6,8±0.16	6,6±0.07	6,4±0.10

Surface morphology analysis

Fig. 5 A1-C1 shows the surface structure of the stainless-steel archwire after immersion in artificial saliva for 1, 7, and 14 d. After one day of immersion (A1), the surface of the wire was still smooth, and only a few small scratches were present on the surface. After 7 d of immersion (B1), the wire surface displayed scratches and small cracks. After 14 d of immersion, the wire surface (C1) exhibited larger and numerous cracks.

Fig. 5 A2-C2 shows the surface structure of the stainless-steel archwire after immersion in 0.2% chlorhexidine for 1, 7, and 14 d. After 1 d of immersion (A2), the wire surface showed deep scratches and cracks; after 7 d (B2), the wire surface showed several cracks and a deep pit; and after 14 d (C2), scratches and large pits with several small pits were observed on the surface of the wire.

Fig. 5 A3-C3 shows the surface structure of the stainless-steel archwire after immersion in 1% *Piper betle Linn* mouthwash for 1, 7, and 14 d. After one day of immersion (A3), cracks were observed in the wire; after 7 d (B3), cracks and several small pits were detected on the wire surface; and after 14 d (C3), cracks and pits were observed on the wire surface.

Fig. 5 A4-C4 shows the surface structure of the stainless-steel archwire after immersion in 2% *Piper betle Linn* mouthwash for 1, 7, and 14 d. After one day of immersion (A4), certain scratches and a few small cracks were observed; after 7 d (B4), several deep cracks were observed on the wire; and after 14 d (C4), scratches and small deep pits were observed on the wire.

Fig. 5 A5-C5 shows the surface structure of the stainless-steel archwire after immersion in 3% *Piper betle Linn* mouthwash for 1, 7, and 14 d. After one day of immersion (A4), the surface of the wire appeared smooth, with a few scratches that were not too deep. After 7 d (B4), several cracks were observed on the wire, while after 14 d (C4), cracks and several small pits that were not too deep were observed.

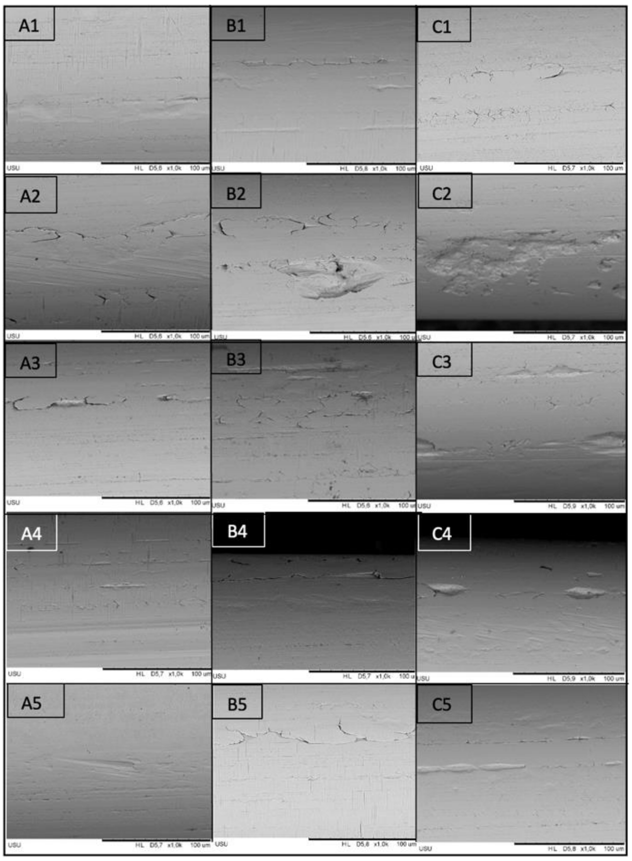

Fig. 5: Surface Morpholgy of wire after immersion (1000x magnification) in artificial saliva (A1-C1), 0.2% chlorhexidine (A2-C2), 1% *Piper betle Linn* mouthwash (A3-C3), 2% *Piper betle Linn* mouthwash (A4-C4), 3% *Piper betle Linn* mouthwash (A5-C5); (A) 1 d, (B) 7 d, (C) 14 d

DISCUSSION

In the oral cavity, orthodontic wire will be exposed to various oral conditions such as an acidic environment, temperature, and saliva, leading to corrosion [10, 11]. Corrosion is defined as damage of orthodontic wire caused by chemical reactions between the metal and the oral cavity component, such as saliva, normal flora, temperature, and acidity. Corrosion that occurs on orthodontic wire reduces the physical and mechanical properties of the wire, thereby increasing the risk of orthodontic treatment failure. In addition, corrosion also affects personal health, such as allergic, mutagenic, and carcinogenic reactions [12]. The process of orthodontic wire corrosion *of stainless steel*, which is in the oral cavity, will release the ions contained in the wire, such as Fe, Cr, and Ni [13]. Metal ion release could affect the mechanical properties of the wire. One of them is wire's stiffness. Stiffness is the amount of force required to bend or flex the wire. Stiffer orthodontic wires are necessary at intermediate stages of orthodontic treatment. Wire stiffness affects the movement of the teeth [14].

The results of this study showed significant differences in stainless steel orthodontic wires in artificial saliva, 0.2% chlorhexidine, 1%, 2%, and 3% *Piper betle Linn* mouthwash for 1, 7, and 14 d; there was an increased Ferrum ion release value and a proportional decrease in stiffness with the rising immersion time. These results are also consistent with the study of Trisnawaty *et al.* 2023, which showed a significant difference in the number of nickel ion released in stainless steel orthodontic brackets immersed in 25% *Piper betle Linn* leaf extract, *Piper betle Linn* leaf extract combined with artificial saliva, and artificial saliva [15]. This result is inversely related to the stiffness study by Sheibaninia *et al.* 2018, who stated that no significant decrease in stiffness was observed in the stiffness of Ni-Ti wires with different cross-sections from the pre-treatment stage to the first and second month of treatment during different tests [16].

The result of this study shows the highest mean value of Ferrum ion (Fe) release from orthodontic wire stainless *steel*, and the lowest stiffness value occurs with 0.2% chlorhexidine, followed by 1%, 2%, and 3% *Piper betle Linn* mouthwash, artificial saliva. The results of this study relevant with Angeline *et al.* 2021, which showed that straight orthodontic wire stainless *steel* rectangular, which immersed in 0.2% chlorhexidine, caused the highest release of nickel ions, followed by *Morinda citrifolia* L. mouthwash, and artificial saliva [17]. This occurs because the acid contained in chlorhexidine mouthwash has high H^+particles and increases when it reacts with metal, so the corrosion rate becomes faster. Acidic conditions will increase $H^+$and form a cathode reaction (reduction) 2 H^+(aq)+2e (aq) → H_2 (g). The reduced H^+ions will bind with electrons released from the oxidation reaction of metal ions and cause corrosion [17, 18].

Piper betle Linn have flavonoid and tannin as phenolic compounds. Phenolic compounds are important secondary metabolites due to their hydoroxyl groups confer scavenging ability [19]. As an antibacterial, flavonoid would inhibit the synthesis of nucleic acid, cytoplasmic membrane, and bacteria metabolism energy. Tannin had bacteria cell-inhibiting properties by denaturing cell protein. Other content saponin could cause protein and enzyme leakage in cells [20]. Beside the antibacterial properties, tannin could inhibit the release of wire metal ions by forming a passive layer on the metal surface. The hydroxyl groups in tannins will react with nickel ions to form more complex compounds. The reaction formed by bonding hydroxyl groups with nickel ions resulted in $Ni(OH)_2$. The passive layer of $Ni(OH)_2$ will create passive conditions on the wire surface, thereby increasing resistance to ion release and preventing a decrease in wire stiffness [21].

Among the treatment groups in this study, there was the lowest ion release and the highest stiffness value in the 3% *Piper betle Linn* immersion group compared to 1% *Piper betle Linn* and 2% *Piper betle Linn*. It because a higher concentration of *Piper betle Linn* would increase tannin and flavonoid compounds, leads to more inhibitor molecules adsorbed on the metal surface. Tannin and flavonoid compounds in *Piper betle Linn* may be responsible for antioxidant activity. Tannins are one of the organic compounds that have the potential to be corrosion inhibitors. Tannins formed complex compounds with Ferrum ions into Fe-titanate on metal surfaces. This Fe-tanate complex compound will become a barrier and coat the metal so that there is no direct contact between the solution and ferrous metal (Fe). In addition, flavonoids in *Piper betle Linn* are compounds that have a chemical structure of C_6-C_3-C_6. Regarding molecular structure, flavonoid compounds have free electron pairs and double bonds as a medium for inhibitors to react with ferrous metals. Flavonoids can also capture free radicals by freeing hydrogen atoms from their hydroxyl groups, which are said to be antioxidant compounds. Therefore, the higher tannin content and the increase in concentration causes the lower release of ferrum ions and decreases wire stiffness [21, 22].

In this study, for 1, 7, and 14 d, there were significant differences in Ferrum ion release and wire stiffness values in all immersion groups. The morphological surface images also revealed intense structural changes—formation of cracks and pits—in the wire during immersion for 1, 7, and 14 d, with an increase in the immersion time. These results are consistent with those of Pastor *et al.* 2023, who stated that the release of Fe, Cr, and Ni ions and surface morphological changes in stainless-steel orthodontic wire immersed in mouthwash containing sodium fluoride for 1, 4, 7, and 14 d increased as the immersion time increased [10]. In this study, pH of all mouthwash is in standard range based on American Dental Association (ADA) and European Standard of Oral Care Products, between pH 3-10.5. The pH of all immersion groups consistently decreased with the rising of immersion time. The decrease in pH in 1%, 2%, and 3% *Piper betle Linn* could be caused by the decomposition of phenol groups in polyphenolic compounds contained in *Piper betle Linn*. The decomposition of phenol groups can cause an increase in the number of H^+ions so that the pH of the mouthwash decreases [22-24].

CONCLUSION

Changes in ferrum ion release and stiffness values of stainless steel orthodontic wires immersed in artificial saliva, 0.2% chlorhexidine, 1%, 2%, and 3% *Piper betle Linn* mouthwash for 1, 7, and 14 d showed that the longer the immersing time, the higher Ferrum ion release and the smaller stiffness value found. The higher concentration of *Piper betle Linn* mouthwash causes more adsorption of tannin compounds so that more inhibitor molecules are adsorbed on the metal surface, and the release of ferrum ions and also the decrease in stiffness could be prevented.

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AUTHORS CONTRIBUTIONS

All the authors have contributed equally.

CONFLICTS OF INTERESTS

Declared none

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