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The effect of the ethanolic extract of African leaves (*Vernonia amygdalina* Delile) on the corrosion rate and microstructure of stainless steel orthodontic wire

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ABSTRACT

Background: *Stainless steel orthodontic wire is commonly used in orthodontic treatment but is prone to corrosion; therefore, an inhibitor is required.* ***Purpose:*** *This study aimed to evaluate the effectiveness of the ethanolic extract of African leaves (Vernonia amygdalina Delile) as a corrosion inhibitor for stainless steel wires.* ***Methods:*** *A laboratory experiment with a pre- and post-test control group design was conducted. Samples included one negative control (artificial saliva), one positive control (chlorhexidine), and two treatment groups (ethanolic extract of African leaves at 3.125 mg/mL and 6.25 mg/mL), totaling 28 samples. Immersion lasted 7 days at 37°C. Corrosion rates were measured by weight loss, and microstructural changes were analyzed using a scanning electron microscope.* ***Results:*** *The highest average corrosion rate was observed in the chlorhexidine group (0.98 ± 0.58 mils per year [mpy]), whereas the lowest was in the 6.25 mg/mL extract group (−0.04 ± 0.80 mpy). Scanning electron microscope analysis revealed notable microstructural differences: the artificial saliva group showed long scratches scattered across the surface and localized round porosity; the chlorhexidine group exhibited rough scratches and widespread porosity; in contrast, the African leaves groups displayed minor scratches and a thin layer presumed to be protective.* ***Conclusion:*** *The ethanolic extract of African leaves considerably affects corrosion rates and microstructural changes in stainless steel orthodontic wires.*

Keywords: *corrosion rate; ethanolic extract of African leaves; microstructural changes; orthodontic wire; stainless steel*
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INTRODUCTION

The orthodontic appliances used for treating malocclusion broadly consist of removable, functional, and fixed appliances. Fixed orthodontic appliances include brackets, bands, arch wires, and accessories or auxiliaries.[1,2] The arch wire plays a crucial role in orthodontic devices by moving teeth back to their regular positions. Stainless steel orthodontic arch wire is the most commonly used wire in orthodontic treatment today. It contains 71% iron, 17%–22% chromium, 8%–12% nickel, and 0.2% carbon. Stainless steel orthodontic arch wire is preferred because of its good modulus of elasticity, high strength, corrosion resistance, and economical cost.[3,4]

Despite these balanced properties, orthodontic wires can undergo degradation in the oral cavity, leading to corrosion. All types of orthodontic wires are susceptible to corrosion, which is influenced by factors such as temperature changes, oral microflora, enzymes in the mouth, and the pH of saliva that remains in constant contact with the wire.[5] Corrosion results in metal ion release, which may trigger allergic reactions in the oral cavity. The release of metal ions from orthodontic wires alters the surface morphology of the

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wire, causing changes such as cavities, peeling layers, and rough surfaces.[6]

Although corrosion in orthodontic wires is unavoidable, its rate can be reduced. One way to inhibit corrosion is by adding inhibitors. Corrosion inhibitors are chemicals that, when added, effectively reduce the corrosion rate. Inorganic or organic substances can act as corrosion inhibitors. Inorganic substances such as phosphate, chromate, and other heavy metals are now limited because of their toxicity and high production costs. This supports the preference for organic inhibitors, as they are non-toxic and do not harm the oral cavity.[7] A study by Pratiwi et al.[8] conducted a phytochemical screening of African leaf extracts, revealing flavonoids, tannins, and steroids. Another study by Febrianti et al.[9] reported that African leaves contain antioxidants at moderate levels, indicating that the antioxidant compounds in African leaves are effective in inhibiting the action of free radicals. These compounds have the potential to act as natural antioxidants and contribute to forming a protective layer that reduces corrosion rates.[10]

Although research on the release of nickel ions from orthodontic wires after immersion in ethanolic extract of African leaves (*Vernonia amygdalina* Delile) has been conducted, studies on the corrosion rate and microstructural changes in stainless steel orthodontic wires after immersion in ethanolic extract of African leaves (*V. amygdalina*) are still lacking. This study aims to investigate the effect of the ethanolic extract of African leaves (*V. amygdalina*) on the corrosion rate and microstructure of stainless steel orthodontic wire.

MATERIALS AND METHODS

This research was a laboratory experimental study with a pre- and post-test control group design, where measurements were taken before and after treatment. The samples consisted of stainless steel orthodontic wire from (American Orthodontics, 3254 Washington Avenue, Sheboygen, USA 53081) with a diameter of 0.016 × 0.022 inches and a length of 3 cm for each wire. A total of 28 samples were divided into four groups. Group A contained seven samples in which the stainless steel orthodontic wire was immersed in artificial saliva. Group B contained seven samples immersed in 0.2% chlorhexidine mouthwash. Groups C and D each contained seven samples immersed in ethanolic extract of African leaves (*V. amygdalina*) at concentrations of 3.125 mg/mL and 6.25 mg/mL, respectively.

The research procedure began with the preparation of 28 pieces of stainless steel wire, each cut to a length of 3 cm, divided into four groups, and weighed. Each wire was weighed three times to obtain the average weight before immersion in artificial saliva, chlorhexidine mouthwash, or ethanolic extract of African leaves (*V. amygdalina*). The pH of each solution was recorded before immersion, and the solutions were then allocated to their respective test tubes.

Wire immersion was conducted according to the test group: group A was immersed in artificial saliva, group B in 0.2% chlorhexidine mouthwash, group C in ethanolic extract of African leaves (*V. amygdalina*) at a concentration of 3.125 mg/mL, and group D in ethanolic extract of African leaves (*V. amygdalina*) at a concentration of 6.25 mg/mL. Each group contained seven samples. After 7 days of immersion at 37 °C, the test tubes were removed from the incubator, and the samples were separated from the solutions. The pH of each solution was measured again after immersion using a pH meter.

After immersion, the wires were carefully removed, thoroughly rinsed with running water, and dried. Once dried, each wire was weighed three times to determine the average post-immersion weight, and the results were recorded for analysis. The corrosion rate, expressed in mils per year (mpy), was calculated using the following formula[10]:

$$Corrotion\ rate = \frac{K \times W}{A \times T \times D}$$

After the wire corrosion rate was determined, the findings were recorded. The measurement of inhibitor efficiency was performed using the following formula[11]:

$$Ef = \frac{Ro - Ri}{Ro} \times 100$$

Subsequently, the wire surface was analyzed using a scanning electron microscope (SEM). The SEM testing instrument was prepared before the sample examination. The wire was cut to a length of 0.5 cm using cutting pliers. The dried sample was then placed on the stage and inserted into the SEM instrument (Model EVO MA 10, Carl Zeiss, Germany). The sample was tested, and the resulting images were displayed on the monitor screen.

RESULTS

The research results indicate that the highest average corrosion rate was observed in group B, where stainless steel orthodontic wire was immersed in 0.2% chlorhexidine mouthwash, with a value of 0.98 ± 0.58 mpy. The lowest average corrosion rate was recorded in group D, where stainless steel orthodontic wire was immersed in ethanolic extract of African leaves (*V. amygdalina*) at a concentration of 6.25 mg/mL, with a value of −0.04 ± 0.80 mpy. Table 1 presents the average corrosion rates of stainless steel orthodontic wire after immersion in artificial saliva, chlorhexidine mouthwash, and ethanolic extract of African leaves (*V. amygdalina*) at concentrations of 3.125 mg/mL and 6.25 mg/mL.

Normality testing was performed using the Shapiro–Wilk test because the total sample size was ≤50. The mean corrosion rate measurements for each group showed the following significance values: group C, p = 0.190 (≥0.05); group D, p = 0.430 (≥0.05); group A, p = 0.704

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(≥0.05); and group B, p = 0.073 (≥0.05). The Shapiro–Wilk test results indicate that all treatment groups met the normality assumption because all significance values were p ≥ 0.05.

Since the data were normally distributed, a homogeneity test was conducted using Levene's test, yielding a significance value of p = 0.056 (≥0.05), which confirms that the homogeneity assumption was met. With all test assumptions fulfilled, a one-way analysis of variance was performed (Table 2).

The results in Table 2 show a significance value of p = 0.04 (< 0.05), indicating a statistically significant reduction in corrosion rate and supporting the inhibitor's effectiveness. This finding demonstrates a substantial difference between the mean corrosion rates of stainless steel orthodontic wires in the control group and those in the treatment groups. The results of the effectiveness test of the ethanolic extract of African leaves (*V. amygdalina*) at concentrations of 3.125 mg/mL and 6.25 mg/mL on the corrosion rate of stainless steel orthodontic wire are presented in Table 3.

The data in Table 3 indicate that the ethanolic extract of African leaves (*V. amygdalina*) was most effective at a concentration of 6.25 mg/mL, achieving an inhibitor effectiveness of 104.3%. At the lower concentration of 3.125 mg/mL, the effectiveness decreased to 93.11%. These findings suggest that the ethanolic extract of African leaves (*V. amygdalina*) can serve as a mouthwash-type corrosion inhibitor during fixed orthodontic treatment. Subsequently, an SEM test was performed to examine microstructural changes on the surface of stainless steel orthodontic wire after immersion in artificial saliva, chlorhexidine solution, and ethanolic extract of African leaves (*V. amygdalina*) at concentrations of 3.125 mg/mL and 6.25 mg/mL.

DISCUSSION

Corrosion can cause oxidation reactions on stainless steel orthodontic wires, leading to the release of free electrons and metal ions. This phenomenon is influenced by various intrinsic and extrinsic factors, including the type of alloy used, pH level, immersion time, and temperature. Because it is difficult to develop a perfectly stable alloy, corrosion of orthodontic wires is inevitable. Therefore, inhibitors are required to slow the corrosion rate. These inhibitors act by forming a thin protective (passive) layer that shields the metal surface.[12] In this study, African leaves were used as a corrosion inhibitor because they contain flavonoids and tannins, which can act as antioxidants. Antioxidants are known to inhibit oxidation activity in the corrosion process.[12]

The highest corrosion rate was observed in wires immersed in the 0.2% chlorhexidine group. This can

Table 1. The average corrosion rate of stainless steel orthodontic wire

Groups (n = 7)	Mean corrosion rate of stainless steel orthodontic wire (mpy) (mean ± standard deviation)
A	0.13 ± 0.42
B	0.98 ± 0.58
C	0.067 ± 0.93
D	−0.04 ± 0.80

Notes:
(+) A positive value indicates a reduction in the weight of the stainless steel orthodontic wire before and after immersion.
(-) A negative value indicates an increase in the weight of the stainless steel orthodontic wire before and after immersion.

Table 2. One-way analysis of variance results for the corrosion rate of stainless steel orthodontic wire

Groups	Mean corrosion rate of stainless steel orthodontic wire (mpy)	Standard deviation	*p*
A	0.13	0.42	0.04*
B	0.98	0.58	
C	0.067	0.93	
D	−0.04	0.80	

*Significant difference (p < 0.05)

Table 3. Inhibitor effectiveness on the corrosion rate of stainless steel orthodontic wire

Groups	Inhibitor effectiveness (%)
C	33.11%
D	94.3%

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be attributed to the higher concentration of H^+ ions in chlorhexidine mouthwash, which increases when reacting with metals and accelerates corrosion. Brar's study reported that the corrosion rate in chlorhexidine mouthwash is higher than in herbal-based mouthwashes. In contrast, the ethanolic extract of African leaves (*V. amygdalina*) showed a lower corrosion rate due to the presence of secondary metabolites, particularly tannins, which function as inhibitor agents. Farmasyanti's research on starfruit leaf extract demonstrated that increasing extract concentration decreases the corrosion rate.[13] The mechanism by which tannins inhibit corrosion involves forming a passive layer on the metal surface. The hydroxyl groups in tannins react with nickel ions to form complex compounds. The reaction between hydroxyl groups and Ni^+ ions produces $Ni(OH)_2$, which acts as a passive layer on the wire surface, reducing the metal's reactivity and increasing its resistance to ion release.[14]

This study also found that increasing the inhibitor concentration correspondingly reduced the corrosion rate of stainless steel orthodontic wires. This reduction is attributed to the tannin content in the ethanolic extract of African leaves (*V. amygdalina*). Tannins contain OH^- groups positioned ortho on the aromatic ring, which form complex compounds with Fe ions, resulting in Fe–tannate. This Fe–tannate complex acts as a barrier, preventing corrosive ions from the external environment from making direct contact with Fe metal.[15] A study on cocoa pod husk extract similarly demonstrated that the highest concentration was most effective in reducing the corrosion rate of stainless steel orthodontic wires.[16] Increasing the inhibitor concentration enhances effectiveness by increasing the number of inhibitor molecules that bind to the wire surface, thereby forming a stronger complex layer that protects the wire surface from corrosive agents.

The pH measurements of the study groups showed that 0.2% chlorhexidine had higher acidity than the other solutions. The acidity level of a solution influences the corrosion rate because higher concentrations of H^+ ions accelerate metal oxidation. It can be concluded that a more acidic pH leads to a higher corrosion rate. H^+ ions undergo reduction by binding with electrons released

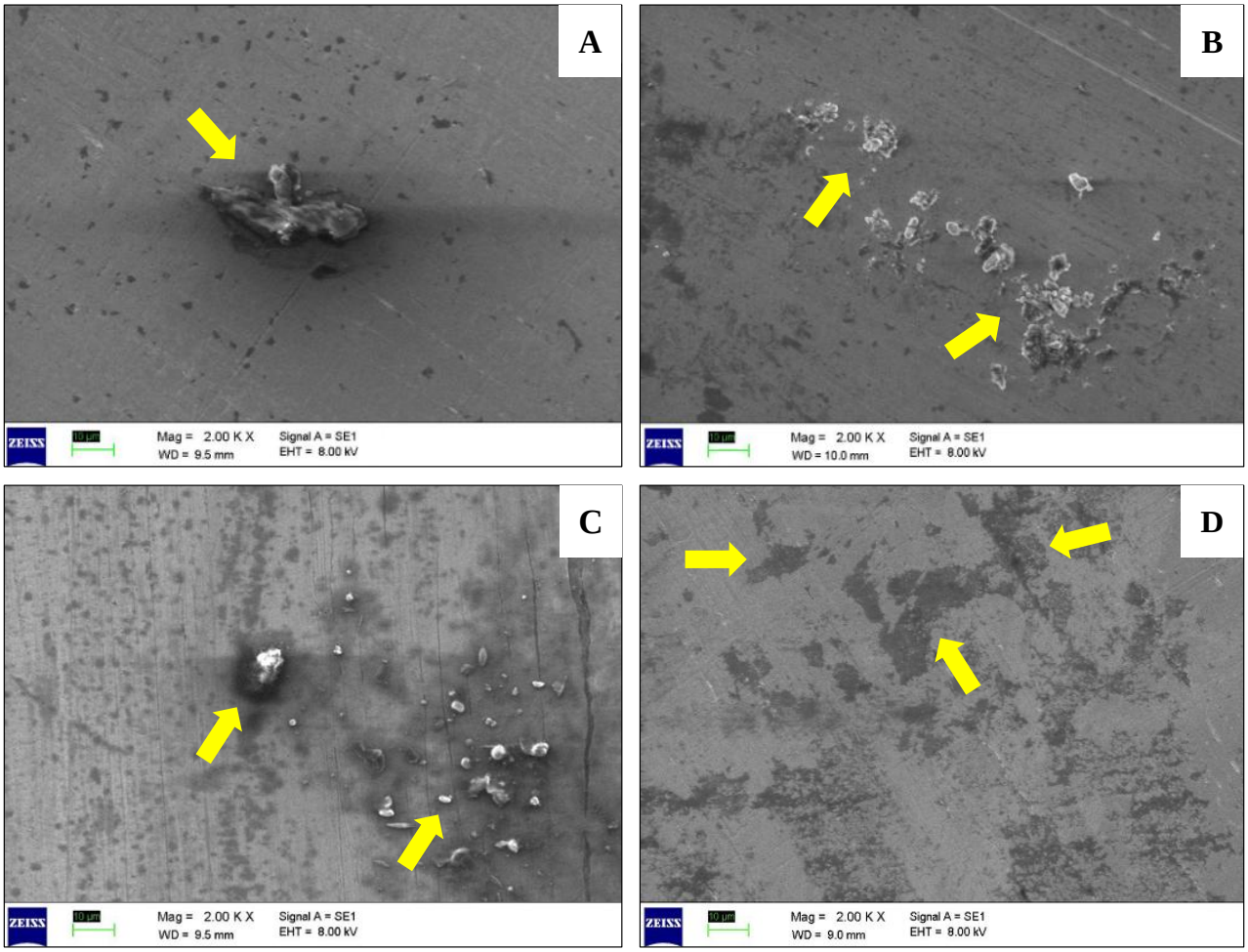

Figure 1. Microstructure of stainless steel orthodontic wire surface at 2,000× magnification following immersion in artificial saliva (pH 9) (A), chlorhexidine mouthwash (pH 6) (B), and ethanolic extract of African leaves (*Vernonia amygdalina* Delile) at concentrations of 3.125 mg/mL (pH 11) (C) and 6.25 mg/mL (pH 10) (D).

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during the oxidation of metal ions. The greater the number of H^+ ions, the higher the reduction rate of H^+, thereby promoting corrosion in acidic environments.[17] The bioactive flavonoid components of the ethanolic extract of African leaves (*V. amygdalina*) may further prevent corrosion by binding free electrons on the metal surface, thereby inhibiting oxidation. When the passive layer covering the metal surface becomes more extensive, the corrosion rate decreases and the inhibitor's effectiveness increases.

Microstructural changes in stainless steel orthodontic wires were observed after immersion in artificial saliva (pH 9), chlorhexidine mouthwash (pH 6), and ethanolic extract of African leaves (*V. amygdalina*) at concentrations of 3.125 mg/mL (pH 11) and 6.25 mg/mL (pH 10). Scanning electron microscopy revealed distinct differences in surface structure between treated and untreated wires. Wires immersed in artificial saliva showed elongated scratch marks spread across the surface and localized round porosities (Figure 1A). Scanning electron microscopy evaluation of as-received stainless steel orthodontic wires at 2,000× magnification revealed a structured surface with parallel streaks.[18]

Stainless steel orthodontic wires immersed in chlorhexidine mouthwash with an acidic pH displayed numerous evenly distributed grooves across the wire surface. The grooves were elongated and had a darkened base. Additionally, scratches and porosities were scattered over the surface. According to Pataijindachote et al.[6] study in 2017, stainless steel orthodontic wires immersed in pH 2.5 solutions exhibited SEM images with broader grooves and additional rounded grooves compared with wires immersed in pH 6 solutions.

The surface analysis of wires immersed in 3.125 mg/mL ethanolic extract of African leaves (*V. amygdalina*) showed a surface structure similar to that of the artificial saliva group, with additional smaller rounded grooves scattered across the surface and localized porosities (Figure 1A). Wires immersed in 6.25 mg/mL ethanolic extract of African leaves (*V. amygdalina*) exhibited a less severe surface structure (Figure 1D) than those immersed in artificial saliva, chlorhexidine, or 3.125 mg/mL extract (Figure 1A–C). The wire surface displayed more regular, elongated grooves, closely resembling the surface morphology of unused stainless steel orthodontic wires. The images also revealed a thin layer suspected to be a protective passive layer formed by the inhibitor, although this layer appeared uneven across the metal surface. These findings are consistent with Verma and Khan's (2011) study, in which mild steel wires immersed in soybean leaf extract showed corrosion inhibition in a 0.5 M HCl solution.[19] Scanning electron microscopy images demonstrated the formation of a protective layer on the surface of mild steel due to the adsorption of inhibitor molecules, effectively preventing corrosion.[20]

This study was limited to a single type of stainless steel orthodontic wire, which may yield different results if other wire types are tested. Furthermore, variations in immersion time may also influence the corrosion rate of orthodontic wires. These findings suggest that the ethanolic extract of African leaves (*V. amygdalina*) considerably inhibits corrosion by forming a protective passive layer, making it a potential natural corrosion inhibitor for stainless steel orthodontic wires.

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