

EFFECTIVENESS OF OOLONG TEA LEAF EXTRACT AS A CORROSION INHIBITOR FOR ORTHODONTICS STAINLESS STEEL WIRE
(EFEKTIVITAS EKSTRAK TEH OOLONG SEBAGAI BAHAN INHIBITOR KOROSI PADA KAWAT ORTODONTI STAINLESS STEEL)

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ABSTRACT

Stainless steel orthodontic wire is the type most often used in the oral environment for long periods, which can potentially generate corrosion. The occurrence of corrosion will cause a hypersensitivity reaction caused by the wire components interacting with the oral environment. One of the efforts that can be used to reduce the corrosion rate is to use organic inhibitors containing antioxidants found in oolong tea leaves. This study aimed to determine the effectiveness of oolong tea leaves as a corrosion inhibitor on stainless steel orthodontic wires. This study is a laboratory experimental study with a post-test-only control group design using stainless steel orthodontic wire with a length of 4 cm and a diameter of 0.41 mm in as many as 24 samples. The samples were divided into four groups; one treatment group was artificial saliva, and

the other three groups were a mixture of artificial saliva given oolong tea leaf extract with concentrations of 6.25%, 12.5%, and 25%, respectively. The measurement of the corrosion rate was carried out using the weight loss method. Then, the orthodontic wire was characterized after immersion using a Scanning Electron Microscope (SEM). The results of the data were analyzed using the Kruskal-Wallis test. The results showed that the highest corrosion rate was found in the artificial saliva group that was not treated at all, and the corrosion rate with treatment decreased. The group with oolong tea leaf extract with a concentration of 25% showed the best results in inhibiting the corrosion rate of wire. In conclusion, oolong tea leaf extract has proved to be an effective corrosion inhibitor for stainless steel orthodontic wire.

Keywords: corrosion; oolong tea leaf; organic inhibitors; saliva; stainless steel

ABSTRAK

Kawat ortodonti stainless steel adalah jenis kawat yang paling sering digunakan dalam jangka waktu lama di lingkungan rongga mulut, sehingga dapat berpotensi mengalami korosi. Terjadinya korosi akan menimbulkan reaksi hipersensitivitas yang disebabkan karena komponen kawat yang berinteraksi dengan lingkungan rongga mulut. Salah satu upaya yang dapat digunakan untuk mengurangi laju korosi adalah dengan menggunakan inhibitor organik yang mengandung zat antioksidan yang terdapat pada daun teh oolong. Tujuan penelitian ini adalah untuk mengetahui efektivitas daun teh oolong sebagai inhibitor korosi pada kawat ortodonti stainless steel. Penelitian ini merupakan penelitian ekperimental laboratorium dengan rancangan penelitian post-test only control group design dengan menggunakan kawat ortodonti stainless steel dengan panjang 4 cm dan diameter 0,41 mm sebanyak 24 buah sampel. Sampel dibagi menjadi 4 kelompok perlakuan yaitu, kelompok saliva artifisial dan 3 kelompok ditambah ekstrak 6.25%, 12.5%, dan 25%. Pengukuran laju korosi dilakukan dengan

menggunakan metode weight loss dan selanjutnya dilakukan karakterisasi kawat ortodonti dengan menggunakan Scanning Electrone Microscope (SEM). Hasil data dianalisis dengan menggunakan uji Kruskall Wallis. Hasil laju korosi tertinggi terdapat pada kelompok saliva artifisial tidak diberi perlakuan sama sekali, dan laju korosi dengan perlakuan mengalami penurunan. Kelompok dengan ekstrak daun teh oolong dengan konsentrasi 25% menunjukkan hasil terbaik dalam menghambat laju korosi kawat. Kesimpulannya adalah ekstrak daun teh oolong terbukti efektif sebagai inhibitor korosi kawat ortodonti stainless steel.

Kata kunci: *daun teh oolong; inhibitor organic; korosi; saliva; stainless steel*

INTRODUCTION

Malocclusion is a term used to describe dental anomalies and occlusal characteristics that represent deviations from ideal occlusion. Furthermore, malocclusion can cause concerns related to dental health and quality of life issues related to dental and oral health, including appearance, function, and psychosocial impacts.¹ Malocclusion is a major dental and oral health problem in Indonesia since it is in third place after dental caries and periodontal disease.² Orthodontic treatment has been developed to assist objective and systematic evaluation of the potential risks to dental health caused by malocclusion.¹

There are several components in orthodontic treatment, including an

orthodontic brace. The brace will interact with the bracket, affecting the teeth' movement and the dental arch's shape, which works by using active force to move the teeth to the desired position.³ Stainless steel orthodontic braces are iron-based steel alloys containing less than 1.2% carbon. When chromium (12–30%) is added to steel, the alloy is referred to as stainless steel, where the chromium works passively, so it is resistant to stains and corrosion.⁴ Stainless steel has several advantages, such as cheapness, biocompatibility, excellent formability, the ability to be soldered and welded, and adequate springback. However, stainless steel also has disadvantages; when it interacts with the oral environment, such as saliva, food and

drinks, changes in temperature, and microflora, it can trigger corrosion conditions or release metal elements making up the alloy.^{5,6} Corrosion is the damage or degradation of metals due to redox reactions between a metal and various substances in its environment that produce unwanted compounds.⁷ Furthermore, corrosion causes loss of material content, structural characteristics changes, or structural integrity. Corrosion on stainless steel orthodontic brace occurs due to the salivary content, which has inorganic components, such as sodium, potassium, chloride, bicarbonate ions, calcium, magnesium, hydrogen phosphate, thiocyanate, and fluoride.^{7,8} The process of corrosion of stainless steel orthodontic braces in the oral cavity can release metal ions in the brace. Metal ions released in the oral cavity, especially Cr and Ni metal ions, can harm health by causing allergic reactions and toxic and cariogenic effects in the human body.⁹ In addition, increased corrosion can cause roughness on the surface of stainless steel orthodontic braces, and it causes the braces to break easily, thus extending treatment time.¹⁰

Corrosion can be slowed down by using chemicals called inhibitors, which form a protective layer on the metal surface. Corrosion inhibitors can come from organic and inorganic compounds containing

groups with lone pairs of electrons, such as nitrites, chromates, phosphates, urea, phenylalanine, imidazoline, and amine compounds. However, these synthetic chemicals are dangerous, the price is relatively high, and they are not environmentally friendly.¹¹ The addition of corrosion inhibitors by using certain substances can reduce the rate of metal corrosion. One is using organic inhibitors, which are safe for the body.¹² Inhibitors organic compounds contain antioxidant compounds and antioxidant content, such as polyphenols, tannins, alkaloids, saponins, astringent oils, and amino acids. One natural ingredient that contains antioxidants and has the potential as a corrosion inhibitor is oolong tea (*Camellia sinensis*).

Oolong tea (*Camellia sinensis*) is a partially fermented tea. The flavour compounds in oolong tea are catechins, amino acids, and sugars. Catechins are known to be very good for the health of the body, and the content of tannins, saponins, alkaloids, and flavonoids functions as antioxidants.^{13,14} In addition, oolong tea contains non-oxidized and oxidized phenolic compounds. It has been reported that oolong tea contains antioxidant compounds. It is anti-carcinogenic and hypoallergenic, and it can even be used in the prevention of diabetes, obesity, atherosclerosis, and heart disease.¹⁵ This

study aims to determine the effectiveness of oolong tea (*Camellia sinensis*) leaf extract as a corrosion inhibitor on stainless steel orthodontic braces.

METHOD

The study type used was laboratory experimental, with the research design using a post-test-only control group design. This study was conducted at the laboratory unit of testing services and certification, Bogor Agricultural University, and the manufacture of oolong tea leaf extract was conducted at the Herbal Laboratory, Yarsi University, in May-July 2022. Moreover, the research sample was an upper 016 stainless steel orthodontic brace with a diameter of 0.41 mm, immersed in the 4 test groups. Each consisted of 24 samples per group—the artificial saliva group, which was not given an inhibitor in oolong tea extract. The artificial saliva group was given oolong tea extract by 6.25%, the artificial saliva group was given oolong tea extract by 12.5%, and the artificial saliva group was given oolong tea extract by 25%. Tools and materials are measuring cup, beaker glass, rotary evaporators, incubator, Scanning Electron Microscope (SEM), stainless steel brace, artificial saliva, and Oolong tea leaf extract

The study began with preparing oolong tea leaf extract using the maceration

method. Dried oolong tea leaves that had been refined were put into a maceration vessel and then soaked in 96% ethanol to obtain a 100% concentration of oolong tea leaf extract. Moreover, samples that had been mixed evenly were left for five days to settle. The top layer of the sample mixture and 96% ethanol was taken using filter paper. The pulp was macerated with 96% ethanol for two days in a place protected from light and then filtered again. The filtrate that had been obtained was then distilled in a rotary evaporator at 40°C to get a thick extract. To make oolong tea leaf extract with a concentration of 6.25%, you need 6.25 grams of oolong tea leaf extract and 100 ml of distilled water; for a concentration of 12.5%, it took 12.5 grams of oolong tea leaf extract and 100 ml of distilled water, and for a concentration, 25% took 25 grams of oolong tea leaf extract and 100 ml of distilled water.

After making the extract, the orthodontic brace preparation was conducted. 12 upper 016 stainless steel brace with a diameter of 0.41 mm were cut to 4 cm, resulting in 24 brace samples of the same length. Afterwards, each brace was weighed in each treatment group using a digital scale, and the results were recorded. The soaking process was then conducted for each treatment group, which contained six orthodontic braces. Soaking and storage

were undertaken in an incubator for 48 hours. After soaking, each brace was weighed in each treatment group. Then, the results were recorded, and the difference between each brace in each treatment group was determined using the final weight of the brace before soaking and subtracting the initial weight of the brace after washing to get the weight loss.

Furthermore, the effectiveness of the inhibitor was calculated. The inhibitor's effectiveness can be obtained by calculating the percentage decrease in the corrosion rate of the metal by comparing the corrosion rate when it is given the inhibitory substance and without the inhibitory substance.

After calculating the effectiveness of inhibitors, observation was conducted regarding the morphological description of the stainless steel orthodontic brace, which experienced the most significant and negligible corrosion by SEM (Scanning Electron Microscope) testing.

Data analysis was conducted using the Shapiro-Wilk normality test, and the homogeneity test was used using the Levene test. The normality and homogeneity test results were unmet, so it was continued with a parametric test called the Kruskal-Wallis test.

RESULT

The results showed descriptive

values of the corrosion rate of stainless steel orthodontic wires immersed in artificial saliva alone or in the control group, artificial saliva with 6.25% oolong tea leaf extract, artificial saliva with 12.5% oolong tea leaf extract, and artificial saliva with 25% oolong tea leaf extract.

Table 1. Descriptive value of corrosion rate

Group	Mean	Standar Deviation
Artificial Saliva	340.60	102.53
Saliva + 6,25% Extract	320.01	71.47
Saliva + 12,5% Extract	251.16	89.84
Saliva + 25% Extract	203.32	64.03

Table 1 shows that the corrosion rate in the control group, namely artificial saliva immersion, produces the highest average of 340.60. After giving 6.25% oolong tea leaf extract, the corrosion rate decreased by an average of 320.01. Similarly, the administration of 12.5% and 25% oolong tea extract also obtained a decreasing average with a mean of 251.16 and 203.32, respectively. Based on the graph above, it is known that the average corrosion rate has a downward trend as the concentration of oolong tea increases. The lowest average

was found in the 25% oolong tea extract.

Table 2. Value of corrosion rate normality test

Group	Sig Shapiro Wilk	Result
Artificial Saliva	0.185*	Normal
Saliva + 6.25% Extract	0.000	Abnormal
Saliva + 12.5% Extract	0.005	Abnormal
Saliva + 25% Extract	0.000	Abnormal

Shapiro-Wilk test, * p > 0.05

From the results of Shapiro-Wilk normality testing, it is known that the data on the corrosion rate of stainless steel orthodontic wires produce a normal distribution (sig>0.05) only in the control group. At the same time, the other groups are not normal (sig<0.05). It does not meet the parametric test requirements, which require all data to be normally distributed. Thus, a non-parametric test, Kruskal Wallis, is used in this study. Therefore, the data in this study did not meet the normality assumption.

Table 3. Value of corrosion homogeneity test

Data	Levene's Test	
	P value	Result

Corrosion rate of stainless steel 0.583* Homogeneous steel

Levene's Test * p > 0.05 significant

The results of the homogeneity test using Levene's test show that the data of the orthodontic stainless steel wire corrosion is a sig value of Levene's Test > 0.05, which indicates that the data variety is homogeneous.

Table 4. The effectiveness of oolong tea (*Camellia sinensis*) leaf extract extract with concentrations of 6.15%, 12.5%, 25%, and artificial saliva. on stainless steel orthodontic braces

	Treatment	N	Mean Rank	P value
Corrosion Rate	Artificial Saliva	6	16.67	0.019*
	Saliva + 6,25% Extract	6	17.33	
	Saliva + 12,5% Extract	6	8.25	
	Saliva + 25% Extract	6	7.75	
	Total	24		

Kruskal Wallis test, * p < 0.05 significant

Based on the results of the Kruskal Wallis statistical test, it was found that the sig value was 0.019 (sig<0.05). It indicates a significant difference in the corrosion rate of stainless steel orthodontic wires between artificial saliva solution and immersion in

artificial solution plus oolong tea leaf extract with concentrations of 6.15%, 12.5%, and 25%.

Finding which treatment group makes a difference is continued with the Mann-Whitney Test with the following results:

Table 5. The differences oolong tea (*Camellia sinensis*) leaf extract extract with concentrations of 6.15%, 12.5%, 25%, and artificial saliva on stainless steel orthodontic

Treatment	Control	6.25% Extract	12.5% Extract	25% Extract
Artificial Saliva		1.000	0.043*	0.043*
Saliva + 6.25% Extract			0.015*	0.023*
Saliva + 12.5% Extract				0.740
Saliva + 25% Extract				

Mann Whitney test, * p < 0.05 significant

The Mann-Whitney test results provide information on further differences in the average corrosion rate of stainless steel orthodontic wires between immersion in 6.25%, 12.5%, and 25% oolong tea leaf solutions. The results showed a difference in the corrosion rate of up to 1.000 mph

between the control group in the form of artificial saliva and the artificial saliva group plus 6.25% extract, with a higher corrosion rate value of the control group. Compared to the 25% extract concentration, the corrosion rate of the 6.25% concentration resulted in a difference of 0.035 mpy. When the group with 12.5% extract concentration immersion is compared with 25% extract concentration, there is only a difference of 0.023 mpy. However, the corrosion rate of wire immersed in 25% oolong tea leaf concentration is more diminutive than immersion in 12.5% concentration.

The Mann-Whitney test results found a significant difference between the control group and all oolong tea leaf extract immersion groups. Thus, it can be concluded that immersion of stainless steel orthodontic wires in oolong tea leaf extract inhibits the corrosion rate. There was a significant difference between the immersion of wire with 6.25%, 12.5%, and 25% extract. The same was shown in the difference between the concentration of 12.5% and 25%, with a difference of 0.023 mpy. Thus, this indicates that the best concentration of oolong tea leaf extract solution as a corrosion inhibitor for stainless steel orthodontic wire is 25%.

Scanning Electron Microscope (SEM)

Analysis

SEM analysis was conducted to see the characteristics or surface description of stainless steel orthodontic braces that had been soaked to see which surface of the brace experienced the most significant corrosion and the most minor corrosion following the order of the treatment groups (Figure 1).

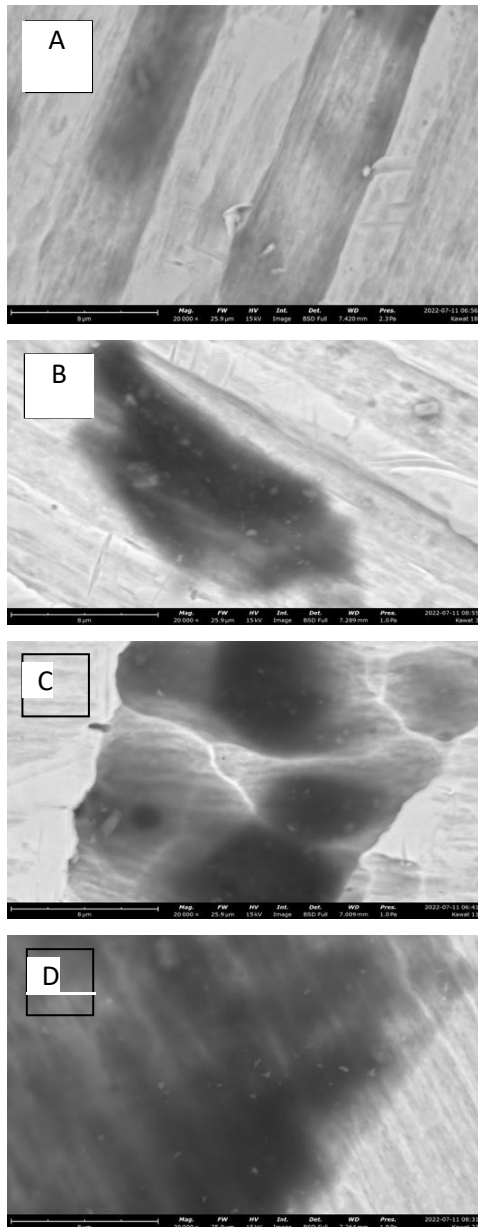


Figure 1. SEM results of brace soaking in artificial saliva without additional inhibitors (A) SEM results of brace soaking in a mixture of artificial saliva and oolong tea leaf extract 6.25% (B) SEM results of brace soaking in a mixture of artificial saliva and oolong tea leaf extract 12.5% (C) SEM results of brace soaking in a mixture of artificial saliva and oolong tea leaf extract 25% (D).

From the samples tested on artificial saliva without additional inhibitors in the form of oolong tea leaf extract, a characteristic description of the orthodontic brace was obtained in the form of several cracks or holes on the surface of the brace. The surface was not covered or coated by oolong tea leaf extract, so the brace was more corroded. In samples soaked only in artificial saliva, 6.7% of chromium and 1.45% of nickel ions were detected. The sample was tested on artificial saliva with the addition of an inhibitor in the form of an oolong tea leaf extract of 6.25%, which obtained a characteristic description of the orthodontic brace where there were several cracks or porous. Still, it appeared that the surface was coated with oolong tea leaf extract, so in samples soaked with the addition of inhibitor 6.25%, a decrease in chromium ions was detected by 3.8% and nickel ions by 1.46%.

The sample tested on artificial saliva

with the addition of an inhibitor in the form of oolong tea leaf extract of 12.5% obtained a characteristic description of the orthodontic brace, which appeared to be a relatively thick crust-coated surface, namely oolong tea leaf extract so that the resulting corrosion product was reduced. Moreover, in the samples soaked with the addition of 12.5% inhibitor, 2.6% of chromium ions and 0.3% of nickel ions were detected. The sample tested on artificial saliva with the addition of an inhibitor in the form of 25% oolong tea leaf extract obtained a characteristic description of the orthodontic brace, the surface of which was coated with oolong tea leaf extract which was quite thick and more extensive so that corrosion was reduced. A 1.5% chromium ion was detected, and a nickel ion was 0.19%.

DISCUSSION

Oolong tea leaves are one of the most widely consumed herbal ingredients that have high antioxidant content, such as tannins, saponins, alkaloids, and flavonoids, where antioxidants will work with amino acids to form complex compounds that are insoluble by metal ions so that they will inhibit the corrosion rate of orthodontic wires. When compared to black tea leaves, oolong tea has a much higher antioxidant content, while compared to black tea,

oolong tea has a lower antioxidant content. Oolong tea also contains non-oxidized and oxidized phenolic compounds with the highest total phenolic content among other types of tea, namely 1.90 mgGAE.¹⁶

The study's results show that the most excellent brace corrosion rate is found in the control group without concentration treatment, with an average corrosion rate of 340.6035953 mpy. Meanwhile, the lowest average brace corrosion rate is found in the group treated with oolong tea leaf extract of 25% with a corrosion rate concentration of 203.3208558 mpy. For the group given the treatment of oolong tea leaf extract with a concentration of 6.25%, the average brace corrosion rate is 320.0098603 mpy, and for the 12.5% treatment group, the average wire corrosion rate is 251.164854 mpy. Furthermore, the average difference between the control group and the oolong extract group at each concentration is a difference of 20.593735 mpy between the control group and the oolong extract group with a concentration of 6.25%. Meanwhile, for the average difference between the control group and the oolong extract group with a concentration of 12.5%, a difference of 89.4387413 mpy is obtained, and the difference between the control group and the oolong extract group with a concentration of 25% shows the results of an average difference of 137.2827395 mpy.

The study results show that the highest corrosion rate occurred in the control group without treatment, and the lowest corrosion rate occurred in the group that had been given the treatment of 25% oolong tea leaf extract. Oolong tea leaves contain compounds like catechins, and amino acids and antioxidant compounds like tannins, saponins, alkaloids, and flavonoids. The content of antioxidants and amino acids can form complex compounds that are almost insoluble by metal ions, allowing these compounds to inhibit the corrosion rate of orthodontic braces. The use of an extract concentration of 25% was conducted since a higher inhibitory concentration will contribute to inhibiting the release of corrosion-causing ions from the metal; the active compound from the extract will work as an inhibitor by forming a passive layer on the metal surface, which will act as a corrosion inhibitor.

The results of this study are in line with research which had been conducted by Roeswahjuni et al. (2019) regarding the effectiveness of green tea extract as a corrosion inhibitor for stainless steel orthodontic brace, where green tea leaf extract with a concentration of 25% is the most effective concentration in inhibiting the corrosion rate of stainless steel orthodontic brace.⁷ Furthermore, the results of this study are in line with research which

had been conducted by Prizty et al. (2020) regarding the effect of the concentration of guava leaf extract (*Psidium guajava L.*) on the corrosion rate of stainless steel orthodontic brace, where the highest efficiency of guava leaf extract inhibitors is found at the largest concentration of extract additions. Guava leaves contain tannins, polyphenolics, flavonoids, monoterpenoids, sesquiterpenes, alkaloids, quinones, and saponins, with a tannin content of 9-12%, which can inhibit corrosion rates, the greater the percentage of extract used, the surface layer of the brace will be closed entirely by inhibitors so that the corrosion rate will be lower.¹⁰

However, it is not in line with the result of the study conducted by Windy et al. (2019), where avocado leaf extract is effective as a corrosion inhibitor at a concentration of 6%, and the corrosion rate increases with more significant extract administration. It might have occurred due to the longer soaking time, which is more than 48 hours. It means that the longer the soaking process, the more corrosion the electrochemical reactions cause, so more corrosion appears, and the ability to inhibit is influenced by the concentration of the extract. In addition, 48 hours is the most effective time for the chemical compounds in the extract to work as inhibitors, which bind to ions that cause corrosion.⁸

The study results showed a decreased corrosion rate in the four types of stainless steel orthodontic wire immersion solutions using alkaline saliva pH with a pH level of 6.8. However, in conditions in the oral cavity, there can be changes in salivary pH due to the influence of food and beverage consumption, temperature changes, and microflora conditions. Because if someone consumes foods and drinks with high acid concentrations, more H⁺ ions from acids will react and undergo reduction. As a result, more and more metal ions are oxidized, thus accelerating the corrosion process. During the corrosion process of stainless steel orthodontic wires, the oxidation rate is proportional to the reduction rate, characterized by the increased release of Ni and Cr ions from the wire.¹⁷

The results of the study show that the highest inhibitor effectiveness is found in soaking of stainless steel orthodontic braces in artificial saliva, which is given 25% oolong tea leaf extract, namely 0.4030582%, and at the addition of 12.5% extract, there is an inhibitor effectiveness of 0.26259016%, and on the addition of 6.25% extract, there is an inhibitor effectiveness of 0.08248722%. Furthermore, in this study, the use of corrosion inhibitors of 6.25%, 12.5%, and 25% is still the optimum concentration, which can reduce the value

of the corrosion rate of stainless steel orthodontic braces. It is supported by the results of calculating the effectiveness of inhibitors, the characteristic description of orthodontic braces through SEM analysis where the brace coated with inhibitors with a concentration of 25% has a reduction in the amount of nickel and chromium ions so that it can be concluded that the best results are obtained at a concentration of 25% oolong tea leaf extract.

CONCLUSION

Based on the research results, it can be concluded that oolong tea leaf extract effectively inhibits the corrosion rate of stainless steel orthodontic wire. Groups with a concentration treatment of 6.25%, 12.5%, and 25% showed a change in inhibiting the corrosion rate of stainless steel orthodontic wire. The most significant change in inhibiting the corrosion rate occurred in the group with a concentration treatment of 12.5% and 25%. However, the group with oolong tea leaf extract with a concentration of 25% showed the best results in inhibiting the corrosion rate of the wire.

CONFLICT OF INTEREST

The author declared no conflicts of interest or potential commercial background in this research.

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