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Electrochemical analysis of corrosion inhibition shallot leaf (*Allium cepa*) extract on mild steel in acidic medium at different immersion times

ABSTRACT

Plant derivatives as eco-friendly corrosion inhibitors are currently greatly interested in much research. Shallot leaf (Allium cepa) was utilized in 0.1M HCl as a corrosion inhibitor of mild steel. The functional groups present in the macerated extract were subsequently identified using an FTIR test. Electrochemical tests such as tafel polarization and EIS were carried out to determine the corrosion inhibition performance of shallot leaf extract on the SS400 steel. The results of the electrochemical analysis show that shallot leaf extract can inhibit mild steel up to 94% at a concentration of 500 ppm. Furthermore, the addition of SLE reduces surface damage of mild steel, which can be seen by SEM.

Keywords: *Corrosion inhibitor, plant extract, shallot leaf extract, carbon steel, immersion time*

1. INTRODUCTION

Mild steel is frequently used as structural steel due to its remarkable mechanical qualities and inexpensive cost. However, it suffers corrosion attacks in acidic environments commonly used in industrial processes such as oil and gas pipelines, chemical processing, paper, textile, refining, etc [1]. Corrosion leads to various losses, including decreased metal service life, safety, human life, cost, and material conservation [2]. Hydrochloric acid (HCl) is a common acid used in the energy industry, particularly oil and gas, for a variety of purposes, including well stimulation, well completion, and well cleaning. It is most often used for acidizing, which is the process of pumping acid into the well to dissolve rock and increase permeability. HCl is also used in well acidizing to remove damage to the wellbore caused by drilling, completion, or production [3].

However, if mild steel is continuously exposed to an environment containing hydrochloric acid (HCl), it will undergo corrosion. Various corrosion inhibitors can be used to stop mild steel from corroding in HCl surroundings. These inhibitors create a protective coating on the metal, blocking any acid from coming into contact with the steel's surface [4].

Inhibitors are generally classified into organic and inorganic, commercially available, and utilized in industrial settings. On the other hand, commercial synthetic inorganic inhibitors are harmful to the environment [5]. Even though organic inhibitors prevent corrosion, many of these substances are created through chemical processes. These technologies have a limited range of applications since they are prohibitively expensive and environmentally harmful [6]. The use of affordable, environmentally friendly chemicals is a hot topic.

Additionally, it has been stated that these compounds work incredibly effectively to safeguard metals in acidic situations [7]. As a result, environmentalists are concerned about the safety of corrosion inhibitors to the environment. Hence, numerous scientists are paying attention to creating green corrosion inhibitors.

Organic inhibitors contain antioxidant compounds that can delay, slow, or stop oxidation by donating electrons. Inhibitors can boost corrosion inhibition with electronegative components such as Oxygen, Sulfur, Nitrogen, and Phosphorus, as well as through the conjugation of double or triple bonds and the presence of aromatic rings [8,9]. Corrosion inhibitors can physically adsorb to the metal

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surface and prevent any interaction with the corrosive solution, or they can chemically react with the surface of the metal to stop corrosion.

Various recent studies have developed a green organic compound that can be obtained from plant extracts such as fruit [10], leaves [11], stem [12], flowers [13,14], seed [15], peel [16] which have shown has high efficiency as a corrosion inhibitor in acidic media. Shallot (*Allium cepa*) has active compounds in flavonoids and phenolic content that capture free radicals to release hydrogen atoms from their hydroxyl groups [17,18]. The highest total flavonoid content is found in shallot leaves when compared to 62 other common plants [19]. The total flavonoid content in leeks of 2720.5 mg/kg ranked first and was followed by Semambu leaves, cayenne pepper, black tea, papaya and guava plants [20]. Flavonoids and phenolics can be used as corrosion inhibitors to protect carbon steel from an aggressive environment and prevent corrosion [21,22]. From this explanation, shallot leaves have the potential to be used as an organic inhibitor. In addition, research has never been conducted on the use of shallot leaf extract (SLE) as an evironmetal friendly corrosion inhibitor. Therefore, this study was conducted to determine the effect of shallot leaf extract on the corrosion phenomenon of mild steel in an acidic solution, namely 0.1 M HCl solution.

2. EXPERIMENTAL SECTION

2.1. Preparation Of Mild Steel

SS400 mild steel was the material chosen for this study, which has the following composition: C:0.19%wt.; Mn: 0.3%wt.; P: 0.009%wt.; S: 0.009%wt.; Si: 0.15%wt.; Ni: 0.11%wt.; Cu: 0.014%wt.; Zn: 0.01%wt.; Al: 0.02%wt.; Fe: Remaining. Mild steel was cut into 10 × 10 × 10 mm^3 dimensions and embedded with resin for the electrochemical experimental procedure. The surface sections of the metals were abraded using sandpapers of various grit levels before each experiment (240, 400, 600, 800, 1000, and 1200).

2.2. Extraction of Shallot Leaves

The extraction of the shallot leaves begins with drying the shallots at room temperature. Shallots are then reduced in size by cutting them. After that, the maceration process was carried out: soaking the shallots in 96% ethanol for four days. The obtained filtrate was then filtered from the residue with filter paper (Whatman No. 44), and the solvent was then evaporated over the course of three days inside an oven at 100°C. The solid waste was then gathered, kept, and employed as an inhibitor (SLE) in several studies in an acidic media.

2.3. Fourier Transform Infrared Spectroscopy (FTIR)

The functional groups contained in the shallot extract were tested using FTIR. The analysis was carried out with the FTIR test instrument. The procedure was performed by homogenizing shallot leaf extract with KBr powder. After that, the mixture is made in pellets or pills and ready for analysis.

2.4. Total Phenolic Content

Total phenol analysis was initiated by making gallic acid master liquor and sample preparation. A master solution of gallic acid was produced by dissolving 1 mg of the acid into 1 mL of methanol (1000 g/mL). Each tube received 25, 50, 100, 150, and 200 L of the master solution by pipetting. To prepare the sample, dissolve 4 mg of the dry sample extract in 4 mL of 1000 g/mL methanol.

The Folin Ciocalteau method with gallic acid as a standard was used to determine the total phenolic content of the extract. The regression equation from the calibration curve obtained was used to represent the total phenolic content of the extract as gallic acid equivalent in mg/g extract. Pipette 25, 50, 100, 150, and 200 mL of Gallic acid standard solutions or a 250 mL sample solution into separate test tubes. Add 7.5 mL of distilled water and 0.5mL of Follin Ciocalteau to each test tube, then shake them. After allowing the mixture to rest for 8 minutes, add 1.5 mL of 20% Na2CO3 to each test tube and mix thoroughly. Set aside for two hours at room temperature before continuing. A wavelength of 765 nm was used to measure absorption. The measurements were carried out twice to ensure the phenol obtained was equal to gallic acid (mg/gr extract).

2.5. Tafel Polarization

Tafel measurements were conducted at 0.6 mV/s in a potential range of -0.25 to +0.25 V, using a GAMRY PCI4G750-50090 potentiostat testing equipment. This electrochemical analysis was completed with a three-electrode cell in the solution, having a platinum counter electrode, calomel reference electrode and a mild steel abrasively prepared working electrode. By extrapolating the Tafel curves, we were able to obtain data such as corrosion potential (E_{corr}), cathodic Tafel slope (β_c) and corrosion current density (i_{corr}). Next, using Eq. 1, the current density of corrosion was applied for determining the inhibition efficiency [15].

$$\eta\ (\%) = \frac{i^0_{corr} - i_{corr}}{i^0_{corr}}\ x\ 100\% \qquad (1)$$

Where

i^0_{corr} is corrosion current density without addition of inhibitor and i_{corr} is current after addition of inhibitor.

2.6. Electrochemical Impedance Spectroscopy

The EIS measurement was conducted with a frequency range between 300,000 Hz to 0.1 Hz using the Gamry instrument, and a Nyquist plot was used to represent EIS data. A fitting line from a practical equivalent circuit measures the impedance parameter. The surface coverage was calculated from corrosion charge transfer resistance using the following equation [15]:

$$\theta\ (\%) = \frac{R_{ct} - R_{ct}^{0}}{R_{ct}}\ x\ 100\% \qquad (2)$$

Where

R_{ct}^{0} is charge transfer resistance without inhibitor and R_{ct} is charge transfer after the addition of inhibitors.

3. RESULTS AND DISCUSSION

3.1. Ftir Analysis

The functional organic compounds that were present in the SLE were identified using the FTIR assay. The results of the FTIR experiment result in a graph that shows tiny peaks in a certain wavelength range, which may indicate the existence of several groups. Figure 1 shows the FTIR results of shallot leaf extract.

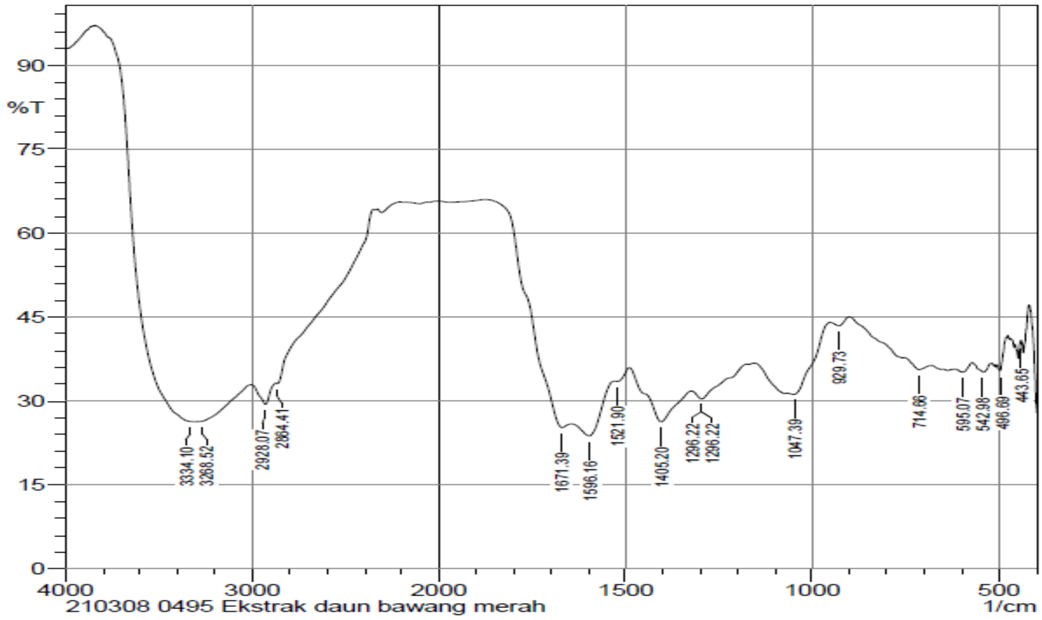


Figure 1. FTIR graph of SLE

Slika 1. FTIR grafik SLE

From the results of this analysis, it can be analyzed for the groups of functions contained in the shallot leaf extract. the functional groups detected in shallot leaf extract are C-H alkyne and alkane and aromatic at 1405.2 cm^{-1}, C-O and C-N bonds carboxyl at 1296.22 cm^{-1}, NO2 and C=C aromatic at 1521.9 cm^{-1}, O-H and N-H groups at 334.1 cm^{-1}[23]. The SLE had N, C, and OH groups, all of which belonged to the alkaloids carboxyl groups, according to the FTIR data. The SLE had polyphenol and flavonoid compounds since they contained aromatic components and phenol.

3.2. Total Phenolic Content

The total phenol content is expressed in EAG (gallic acid equivalent), a unit for defining the number of milligram equivalents of the gallic acid compound from one gram of sample. The total phenol test results showed that the tested shallot leaf extract has a total phenol content of 5.535 mgEAG/g extract.

3.3. Tafel Polarization

Figure 2 depicts Tafel polarization's outcome shallot leaf extract at various immersion times and inhibitor concentrations. The increase of SLE concentration to 500 ppm resulting high inhibition efficiency that reaches 94 percent at 60 min of immersion, as shown in table 2. At 500 ppm corrosion current of SLE decreases from 752.9 μA cm^{-2} to 107.7 μA cm^{-2} (0 min), from 793.3 μA cm^{-2} to 60,90 μA cm^{-2} (30 min), and from 802,4 to 46,74 μA cm^{-2} (60 min). The corrosion current density and corrosion rate of mild steel decreases when concentration of the SLE inhibitor increases, resulting in higher efficiency inhibition. The deposited corrosion inhibitor molecule on the metal surface formed a protective barrier at the metal interface, protecting the metal surface from the acidic media and reducing the metal's corrosion rate [24,25].

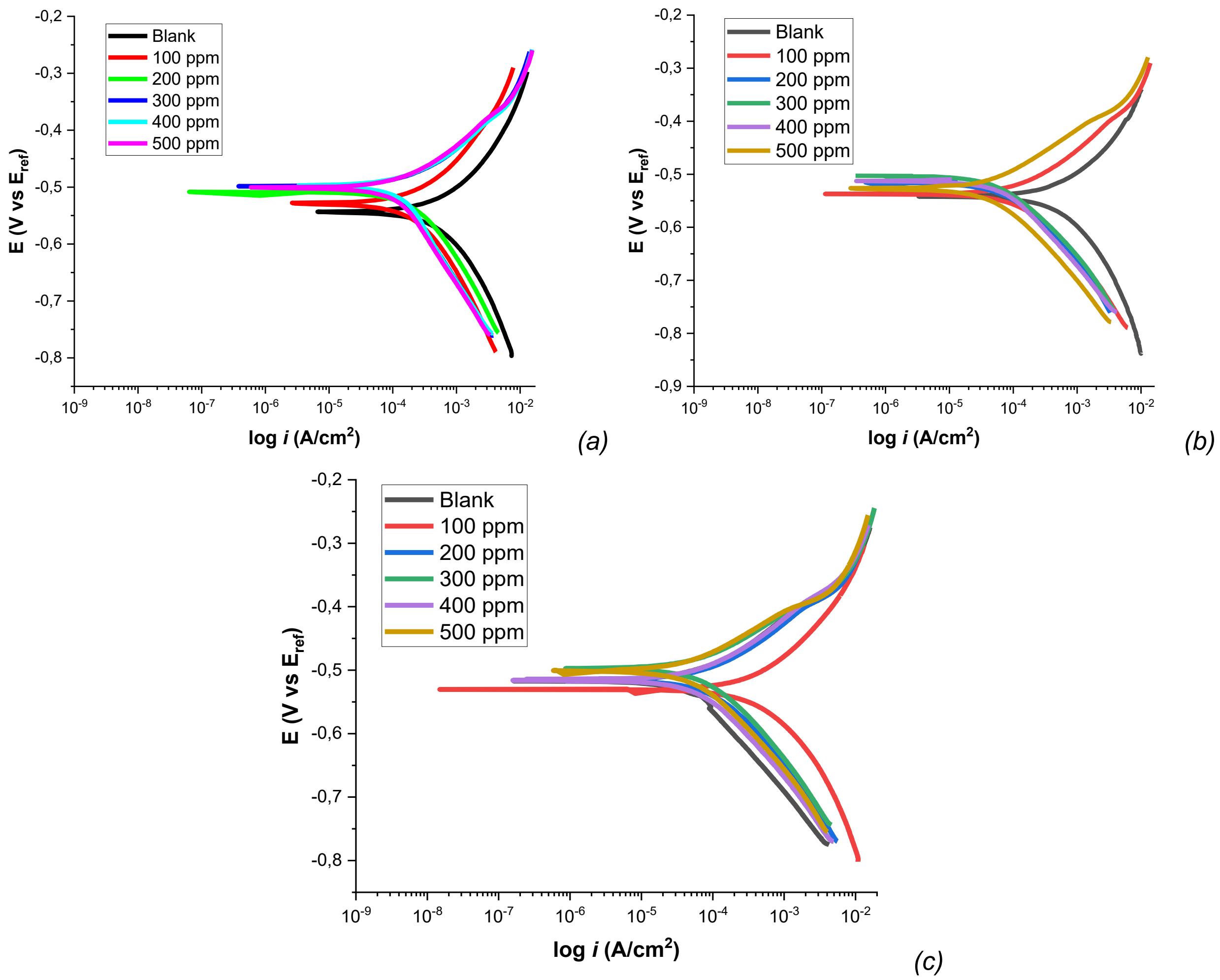


Figure 2. Tafel polarization of mild steel in the absence and presence of SLE in 0.1 M HCl after immersion (a) 0 minutes, (b) 30 minutes, (c) 60 minutes

Slika 2. Tafel-ove krive mekog čelika u odsustvu i prisustvu SLE u 0,1 M HCl nakon potapanja (a) 0 minuta, (b) 30 minuta, (c) 60 minuta

Corrosion potential slightly increases with adding SLE if it is compared to the blank. SLE addition reduces anodic dissolution and blocks oxidation reactions by preventing reaction sites that become active on the steel surface [26], showing that adding inhibitors to the solution reduces both cathodic and anodic currents without modifying the mechanism [27,28]. It has been reported that if the corrosion potential movement from blank to anodic or cathodic directions is more significant than 85 mV is categorized as an anodic or cathodic inhibitor. In this report, the greatest movement of E_{corr} value was 45.5 mV indicating that inhibitor SLE is a mixed-type inhibitor.

Table 1 shows the current density (i_{corr}) and corrosion rate of mild steel decrease after 30 and 60 minutes of immersion. The decrease in current density as well as corrosion rate indicates that SLE has a great inhibitor effectiveness and durability in film inhibition [29]. The inhibitor efficiency rises at the longer immersion time up to 94.2% in 500 ppm of SLE. The immersion period will also give the antioxidant compound molecules of the shallot leaf extract and the SS400 steel enough time to adsorb or inhibit one another and create a film or monolayer layer on the steel surface [30]. Figure 3 examines the influence of concentration on changing the corrosion potential and decreasing current density to reduce corrosion rate and increase inhibition efficiency.

Table 1. Electrochemical parameters of SLE

Tabela 1. Elektrohemijski parametri SLE

Immersion Time (min.)	Concentration (ppm)	β_c (V/decade)	E_{corr} (mV)	i_{corr} (μA/cm^2)	CR (mpy)	η (%)
0	0	0.2347	-544.8	752.9	344.9	0
	100	0.2116	-530.2	279.4	128.0	62.89
	200	0.1932	-508.3	255.1	116.9	66.10
	300	0.1744	-580.8	116.5	53.37	84.52
	400	0.1735	-499.3	112.1	51.36	85.11
	500	0.1717	-503.0	107.7	49.33	85.70
30	0	0.2318	-541.9	793.3	363.4	0
	100	0.1588	-538.5	184.9	84.69	76.70
	200	0.1567	-515.4	70.19	50.96	85.98
	300	0.1441	-501.5	85.55	39.19	89.21
	400	0.1398	-512.7	70.99	32.52	91.05
	500	0.1409	-528.7	60.90	27.90	92.32
60	0	0.2125	-530.4	802.4	367.6	0
	100	0.1486	-515.3	131.3	60.17	83.63
	200	0.1479	-497.5	108.4	49.65	86.49
	300	0.1378	-516.0	77.31	35.42	90.36
	400	0.1365	-500.6	70.71	32.39	91.19
	500	0.1311	-517.1	46.74	21.41	94.17

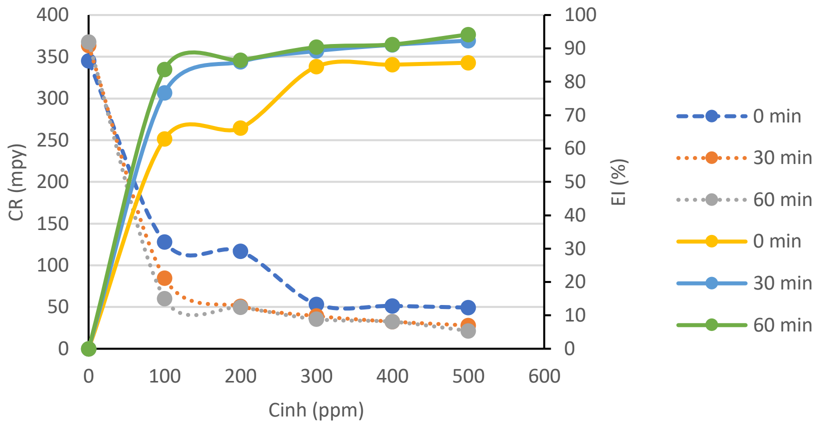


Figure 3. Graph of the effect of concentration on corrosion rate and inhibition efficiency

Slika 3. Grafikon uticaja koncentracije na brzinu korozije i efikasnost inhibicije

3.4. Electrochemical Impedance Spectroscopy (EIS)

The EIS tests create Nyquist curves which to learn more about the phenomena carried on by adding XME inhibitors at the metal/electrolyte contact. EIS test was carried out at various concentrations and immersion periods both without and with the addition of inhibitors. The Nyquist curve is typically depicted as a semicircle, showing the difference between the real and imaginary impedance values. Figure 4 displays the Nyquist curve for this study. EIS data were measured between 300 kHz and 0.1 Hz in frequency. A primary Randles-type electrical circuit will simulate a Nyquist curve derived from the test findings.

The semicircular Nyquist diameter can be assumed as an inhibitor's capacity to prevent corrosion. The larger the curve's radius indicates the greater charge transfer resistance (R_{ct}). Semicircular Nyquist diameter grows more significant with the SLE inhibitor presence and increases as SLE concentration rises. Likewise, increased immersion time will make an enormous semicircular diameter Nyquist curve. The fitting line from EIS was used the Randles model editor, which is shown in Figure 5, to determine the solution resistance (R_u), charge transfer resistance (R_{ct}), and double-layer capacitance (C_{dl}). The resistance parameters of SLE on SS400 steel are shown in Table 3.

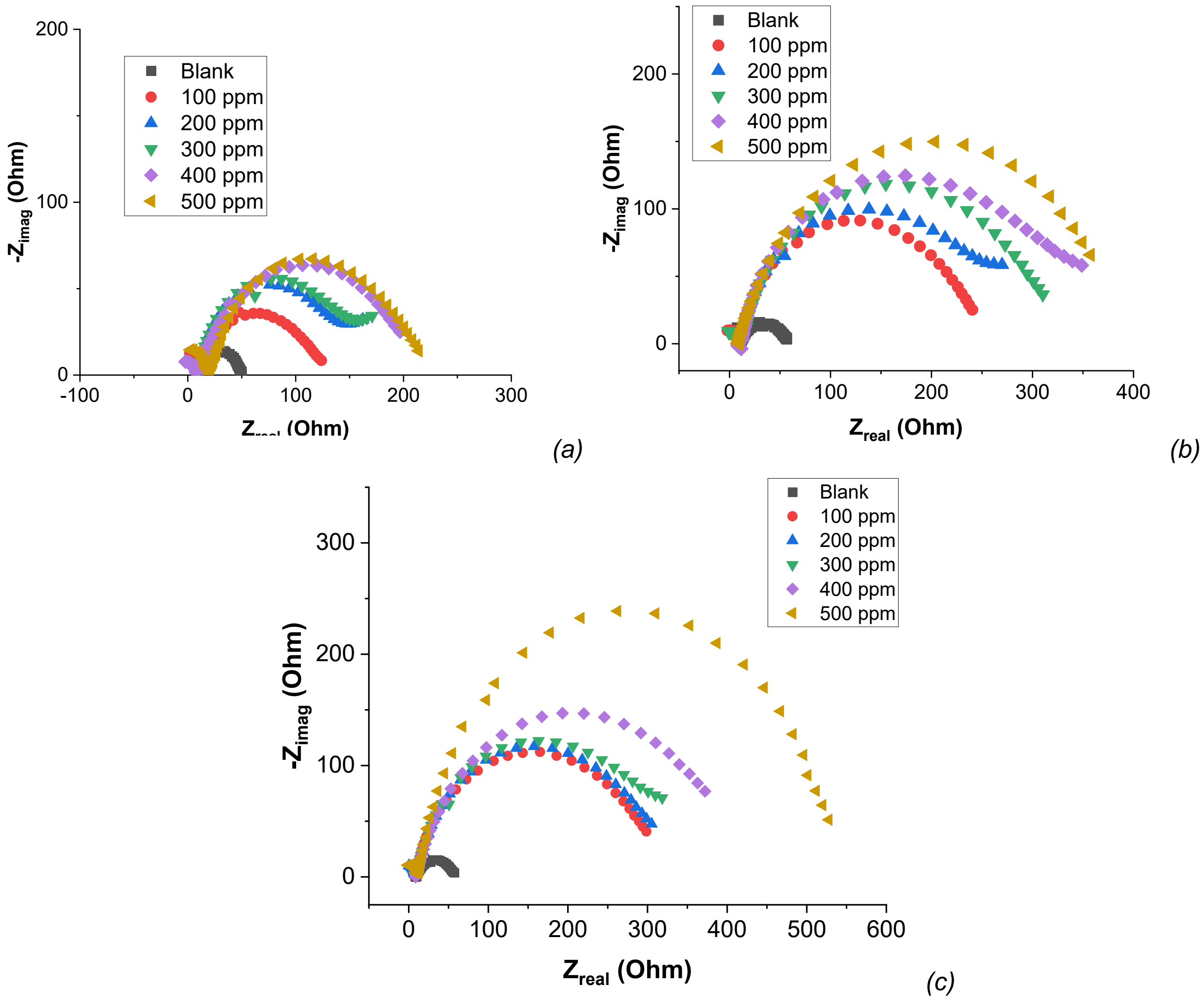


Figure 4. Nyquist plot of mild steel in various concentration of SLE in 0.1 M HCl after immersion (a) 0 minutes, (b) 30 minutes, (c) 60 minutes

Slika 4. Nyquist-ove krive mekog čelika u različitim koncentracijama SLE u 0,1 M HCl nakon potapanja (a) 0 minuta, (b) 30 minuta, (c) 60 minuta

The observation from Table 2 shows that the Rct value increases by increasing SLE concentration and the longer immersion duration of the steel in the solution. Because of resistance in a layer on the electrode's surface, R_{ct} rises, causing a drop in potential and a fall in corrosion rate. After adding SLE, a barrier will form on the metallic surface, preventing electrons or ions from passing. It shows that as the inhibitor concentration rises, inhibitor molecules that adsorb to the surface of the steel increase resulting in increasing the steel's resistance to charge transfer only by inhibiting active sites on the metal [31]. An increase in R_{ct} value affects better inhibition efficiency, calculated using eq.2. Table 2 also shows that increasing immersion increases the R_{ct} value. Excellent surface coverage was obtained after a longer immersion time of 60 minutes, improving the inhibitor's efficiency. This trend suggests that SLE molecules adsorbed to the metal/solution interface create a long-term protective barrier, inhibiting the molecules from adhering to the metal surface and, thus, shielding it from corrosive attack. Plant extracts, including *syzygium cumini* [32], rosa canina [33], esfan seed [34], and dardagan fruit [29] also exhibit this behavior.

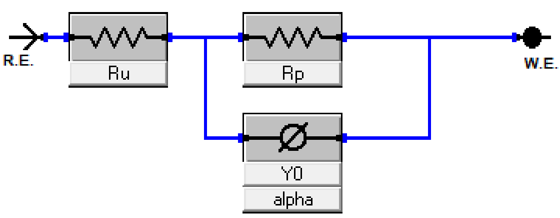


Figure 5. An equivalent circuit model was used in this study

Slika 5. Model ekvivalentnog kola korišćen u ovoj studiji

Table 2. The fitted data of EIS result in concentration and immersion time variations

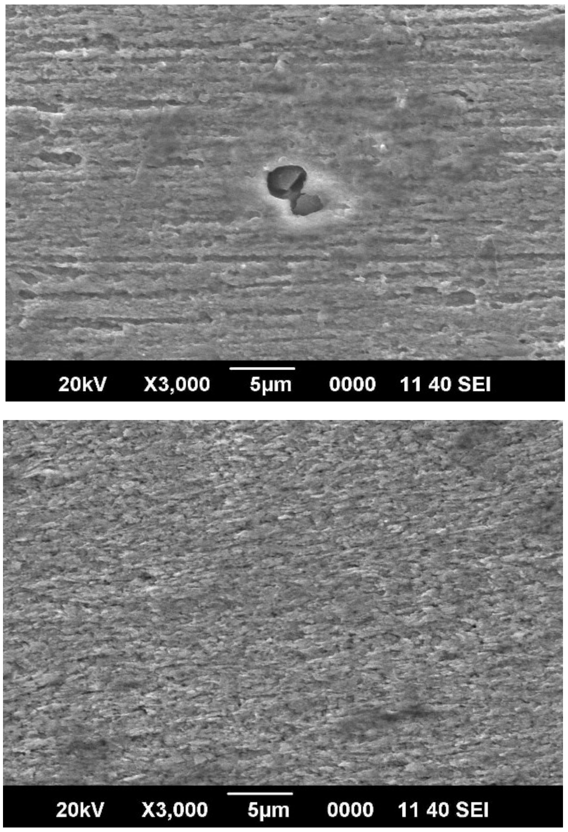
Tabela 2. Dobijeni podaci EIS-a pri varijacijama koncentracije i vremena uranjanja

Immersion Time (minute)	Conc. (ppm)	R_u (ohm)	R_{ct} (ohm)	Y^0 (10^4)	n	C_{dl} (μF)	θ (%)
0	0	9.543	42.14	4.38	0.73	97	0.00
	100	9.807	115.5	3.02	0.72	80	63.52
	200	8.274	153.4	1.24	0.77	37	72.53
	300	8.286	162.1	1.11	0.77	33	74.00
	400	8.272	198.0	1.05	0.75	29	78.72
	500	13.26	210.9	1.18	0.70	24	80.02
30	0	9.252	49.56	6.17	0.69	131	0.00
	100	6.862	244.7	1.52	0.81	70	79.75
	200	8.976	268.8	0.95	0.82	43	81.56
	300	8.029	313,8	0.93	0.81	40	84.21
	400	9.304	346.9	0.80	0.80	33	85.71
	500	9.003	392.2	0.81	0.78	31	87.36
60	0	8.485	51.07	12.05	0.58	401	0.00
	100	9.313	301,6	1.26	0.82	390	83.07
	200	6.466	323.6	1.85	0.79	388	84.22
	300	8.727	330.4	3.82	0.82	382	84.54
	400	9.019	404.0	1.04	0.80	297	87.36
	500	7.404	527.9	0.45	0.92	162	90.33

From Table 2, the results demonstrate clearly that double-layer capacitance (C_{dl}) decreases as R_{ct} values increase. The C_{dl} value gradually decreases as inhibitor concentration increases, showing that the adsorption of corrosion inhibitor leads to the dielectric constant being reduced, thickening the electric double-layer at the metal/electrolyte interface [35].

3.5. Surface Characterization

The surface of corrosion inhibitors of SLE was characterized by SEM. The carbon steel was soaked in 0.1M HCl in the blank and with the addition of 500 ppm SLE for 24 hours at room temperature. Figure 6 shows the smoother surface on the steel was found after adding of 500 ppm of SLE. On the other hand, pitting corrosion can occur and the surface of carbon steel without the presence of SLE seems rough. The rough surface of the metal without inhibitors indicates that the surfaces formed a corrosion product from the interaction between metal and electrolyte. The addition of SLE increases the homogeneity of the surface and minimizes the roughness of the steel substrate. Surface smoothness is produced when a layer of protective Fe^{2+} complex and shallot leaf extract is formed on the metal surface, reducing the carbon steel corrosion rate [36,37].



(a)

(b)

Figure 6. SEM morphology of carbon steel (a) blank and (b) after the addition of 500 ppm SLE

Slika 6. SEM morfologija ugljeničnog čelika (a) blanko i (b) nakon dodavanja 500 ppm SLE

3.6. Proposed Inhibition Mechanism

Potentiodynamic polarization and EIS observed a greater efficiency at higher concentrations of corrosion inhibitors. Thus, the mechanism and efficiency of inhibition are dependent on inhibitor concentration. The difference in immersion time on mild steel also gives another effect on the adsorption of the inhibitor. During the first step of immersion, the plant extract components were adsorbed onto the steel surface. The process can lead to the formation of a barrier layer that protects the metal surface from damaging the HCl environment. The inhibitor molecules keep adhering to the metal surface, producing a thicker protective layer as the immersion duration lengthens. This may result in a decrease in the corrosion rate and an improvement in the effectiveness of the inhibitor. However, due to the aggressive environment and prolonged immersion times, the inhibitor molecules may begin to desorb from the metal surface. This may cause the inhibition efficiency to drop and the rate of corrosion to rise [38]. Figure 7 provides an illustration of the potential SLE adsorption process on the surface of mild steel.

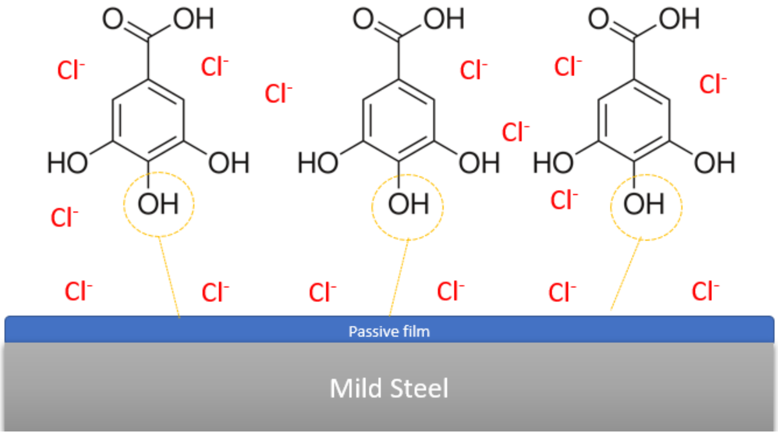


Figure 7. Possible adsorption mechanism process of SLE onto mild steel surface

Slika 7. Mogući proces mehanizma adsorpcije SLE na površini od mekog čelika

4. CONCLUSION

In this study, ethanol extracted an eco-friendly corrosion inhibitor from shallot leaf and evaluated using electrochemical and surface analysis in 0.1 M HCl. Tafel polarization of carbon steel shows a decrease in corrosion rate after the addition of SLE with 94% inhibition effectiveness after 60 minutes of immersion. The polarization test also showed that SLE acted as a mixed-type inhibitor. SEM morphology of metal surface showed that the inhibitor adsorption of SLE significantly decreases the surface damage of mild steel.

Conflict of interest

All of the authors declare there is no conflict of interest.

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IZVOD

ELEKTROHEMIJSKA ANALIZA INHIBICIJE KOROZIJE EKSTRAKTA LISTA SHALLOT *(Allium cepa)* NA MEKOM ČELIKU U KISELOJ SREDINI PRI RAZLIČITIM VREMENIMA POTARPANJA

Biljni derivati kao ekološki prihvatljivi inhibitori korozije su trenutno veoma zainteresantni za mnoga istraživanja. List SHALLOT (Allium cepa) je korišćen u 0,1 M HCl kao inhibitor korozije mekog čelika. Funkcionalne grupe prisutne u maceriranom ekstraktu su naknadno identifikovane korišćenjem FTIR testa. Sprovedeni su elektrohemijski testovi kao što su Tafel polarizacija i EIS da bi se odredio učinak inhibicije korozije ekstrakta lista Shallot na SS400 čeliku. Rezultati elektrohemijske analize pokazuju da ekstrakt lista Shallot može inhibirati meki čelik do 94% u koncentraciji od 500 ppm. Štaviše, dodatak SLE smanjuje površinsko oštećenje mekog čelika, što se može videti pomoću SEM.

Ključne reči: *inhibitor korozije, biljni ekstrakt, ekstrakt lista ljutike, ugljenični čelik, vreme potapanja.*

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