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Inhibition of corrosion of mild steel in simulated oil well water by aqueous extract of *Hibiscus rosa-sinensis* flower

ABSTRACT

An aqueous extract of Hibiscus rosa-sinensis flower (HRF) has been used as corrosion inhibitor in controlling the corrosion of mild steel in simulated oil well water (SOWW). Weight loss method reveals that 10 % v/v of the extract offers 82 % inhibition efficiency (IE) to mild steel (MS) immersed SOWW. The mechanistic aspects of corrosion inhibitive effect have been investigated by polarization study and AC impedance spectra. Polarization study reveals that the mixed type of inhibitor in the presence of inhibitor system. The corrosion inhibition effect is confirmed by the increase in the linear polarization resistance value and decrease in the corrosion current value. The protective layer is formed on the metal surface is confirmed by the AC impedance spectra. This is confirmed by the fact that there is increase in charge transfer resistance value and decrease in the double layer capacitance value. The adsorption of inhibitor molecule obey Langmuir adsorption isotherm. The protective coating is characterized by FTIR spectroscopy. It confirms that the inhibitor has coordinated with ferrous ion the metal surface through the active principle component of β-sitosterol, quercetin and kaempferol-3-xylosylglucoside. The surface morphology has been investigated by scanning electron microscopy (SEM). Hence, aqueous extract of Hibiscus rosa-sinensis flower with simulated oil well water good corrosive inhibitive effect on pipeline made of mild steel.

Keywords: *Hibiscus rosa-sinensis, simulated oil well water, mild steel, corrosion inhibition, surface morphology, SEM*

1. INTRODUCTION

Corrosion normally occurs in oil and gas pipelines. Since the pipelines play the role of transporting oil and gas from the wellheads to the processing facilities, they are exposed to the continuous threat of corrosion, from the date of commissioning up to decommissioning or abandonment [1]. Almost any aqueous environment can promote corrosion, which occurs under numerous complex conditions in oil and gas production, processing and pipeline systems [2]. Internal corrosion in oil and gas industry is generally caused by water, carbon dioxide (CO_2) and hydrogen sulfide (H_2S) and also can be aggravated by microbiological activity [3]. One method used to reduce corrosion in the petroleum industry is corrosion inhibitors. The inhibitors must be put above a specific minimum concentration to obtain an optimal inhibition.

The plant extraction may become corrosion inhibitors due to the advantages of wide sources and biodegradation. It was previously reported that the extracts of *Napoleonaeaimperalis* [4], *Cardiospermumhalicacabum* leaf [5,6], *Tiliacordata* [7], *Morindacitrifolia* [8], *Acanthus montanus* [9] and *Calotropisprocera* [10] showed corrosion inhibition. Heteroatoms such as nitrogen (N), oxygen (O), sulfur (S) and phosphorus (P) can be used as adsorption centers of inhibitor molecules and these atoms also have become important features of efficient corrosion inhibitors. Anuratha et al have used the corrosion inhibition of carbon steel in low chloride media by an aqueous extract of *Hibiscus rosa-sinensis* [11]. Rajendran et al have studied the corrosion behaviour of aluminium in the presence of an aqueous extract of *Hibiscus rosa-sinensis*

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[12], Desai have studied the *Hibiscus rosa-sinensis* leaves extracts used as corrosion inhibitors for mild steel in HCl [13]. However, to the best of our knowledge the aqueous extract of *Hibiscus rosa-sinensis* flower has not been investigated in SOWW medium for its anticorrosion properties.

Hibiscus rosa-sinensis (Figure 1) is a perennial ornamental woody plant; belong to Malvaceae family available throughout India. Various parts of this plant like roots, leaves and flowers have been known to possess medicinal properties like anti inflammatery, antifungal, anti-microbial, oral contra-ceptive, laxative, aphrodisiac, menorrhagicetc [14]. The aqueous extract of *Hibiscus rosa-sinensis* flower contain main active principle constituents such as β-sitosterol, quercetin and kaempferol-3-xylosylglucoside [15].

In the present study, the corrosion inhibitive properties of the aqueous extract of *Hibiscus rosa-sinensis* flower (HRF) in controlling corrosion of mild steel in SOWW solution have been investigated by weight loss and electrochemical study. The HRF extract of active principle constituents have been characterized by FTIR, with the formation of protective film formation against corrosion. The morphology of the mild steel surface was examined by SEM.

Figure 1. Hibiscus rosa-sinensis flower

Slika 1. Cvet Hibiscus rosa-sinensis

2. MATERIALS AND METHODS

Preparation of inhibitor

The aqueous extract of *Hibiscus rosa-sinensis* flower (HRF) were prepared by the method of soxhlet extraction. About 100 g of powdered plant of *Hibiscus rosa-sinensis* flower was uniformly packed into thimble and extracted with 1000 ml of double distilled water. The process of extraction continues till the solvent in siphon tube of the extractor becomes colourless. After the process of extraction, the extract was kept overnight for cooling and made up to 1000 ml with the same double distilled water to get 10 % (w/v) extract.

Preparation of simulated oil well water (SOWW)

In 100 mL of DD water, sodium chloride (3.5 g), calcium chloride (0.305 g) and magnesium chloride (0.186 g) are added. Just before experiment add 0.067 g sodium sulfide and 0.4 mL of concentrated hydrochloric acid to generate hydrogen sulfide gas to form a simulated oil well water containing 100 ppm of H_2S [16].

Preparation of mild steel (MS)

Mild steel specimens (0.0267 % S, 0.06 % P, 0.4 % Mn, 0.1 % C and the rest iron) of dimensions 1.0 cm x 4.0 cm x 0.2 cm were polished to a mirror finish and degreased with acetone.

Weight loss method

Mild steel specimens in triplicate were immersed in 100 ml of the simulated oil well water containing various concentrations of the inhibitor (aqueous extract of *Hibiscus rosa-sinensis* flower) for a period of one day. The weight of the specimens before and after immersion was determined using a Shimadzu balance, model AY62. The corrosion products were cleaned with Clarke's solution [17]. The corrosion rate were calculated using the following equation [18,19].

$$\text{Corrosion rate} = W/AT$$

Where

W = loss in weight (mg)

A = surface area of the specimen (dm^2)

T = period of immersion (days)

The corrosion rate is expressed in mdd units [mdd = mg/(dm^2)(day)]

The inhibition efficiency was calculated using the relation.

$$\text{Inhibition efficiency} = [(CR_1-CR_2)/CR_1] \times 100\ \%$$

Where

CR_1= corrosion rate in the absence of inhibitor

CR_2 = corrosion rate in the presence of inhibitor.

Electrochemical studies

In the present work, corrosion resistance of MS immersed in various test solutions were measured by polarization study and AC impedance spectra.

Polarization study

Polarization studies were carried out in a CHI-electrochemical work station with impedance model

660A. It was provided with iR compensation facility. A three electrode cell assembly was used. Mild steel was used as working electrode, platinum as counter electrode and saturated calomel electrode (SCE) as reference electrode. From polarization study, corrosion parameters such as corrosion potential (E_{corr}), corrosion current (I_{corr}), Tafel slopes, anodic=b_a and cathodic=b_c and linear polarization resistance (LPR) value were calculated.

AC Impedance spectra

The same instrument and set-up used for polarization study was used to record AC impedance spectra also. A time interval of 5 to 10 min was given for the system to attain a steady state open circuit model. The real part (Z') and imaginary part (-Z") of the cell impedance were measured in Ohms at various frequencies. AC impedance spectra were recorded with initial E(V) = 0, high frequency (1-10^5 Hz), low frequency (1 Hz), amplitude (V) = 0.005 and quiet time (s) = 2. From Nyquist plot the values of charge transfer resistance (R_t) and the double layer capacitance (C_{dl}) values were calculated.

FTIR spectra

FTIR spectra were recorded in a Perkin - Elmer "Spectrum Two" spectrophotometer. The film was carefully removed, mixed thoroughly with KBr made in to pellets and FTIR spectra were recorded.

Scanning electron microscopy (SEM)

The mild steel specimens immersed in various test solutions for one day were taken out, rinsed with double distilled water, dried and subjected to the surface examination. The surface morphology measurements of the mild steel surface were carried out by scanning electron microscopy (SEM) using CAREL ZEISS make model EVO-18.

3. RESULTS AND DISCUSSION

Pipelines made of MS are used to carry oil well water in petroleum industry. These pipes may undergo inner corrosion. To prevent corrosion, corrosion inhibitors are used. The corrosion resistance of MS in SOWW, has been evaluated by polarization study and AC impedance spectra. Surface morphology was analysed by scanning electron microscopy. Aqueous extract of HRF has been used as corrosion inhibitor.

Weight loss method

The corrosion rate of mild steel in SOWW is studied by weight loss method in the absence and presence of aqueous extract of *Hibiscus rosa-sinensis* flower (HRF) on immersion period of one day at 30°C and the percentage inhibition efficiency is calculated. Table 1 shows the variation of corrosion rate (mdd), inhibition efficiency (IE in %) and surface coverage (θ) with varying concentration of inhibitor (in % v/v). From the data, it is observed that corrosion rate is significantly lowered down in the presence of the inhibitor. The corrosion rate is found to be dependent on the concentration of the inhibitor. With the increase in concentration, the corrosion rate decreases gradually. The decrease in the corrosion rate is due to the presence of active phytochemicals.

Figure 2 shows the variation of corrosion rate (CR) with concentration of inhibitor (in % v/v) and the data reveals that inhibition efficiency increases with increase in the concentration of the inhibitor (HRF) in SOWW medium. The maximum inhibition efficiency of 82 % is noticed at 10 % v/v of the inhibitor concentration. The decreasing corrosion rate and increasing inhibition efficiency are attributed to the adsorption of inhibitor on the metal surface.

Table 1. The corrosion rate, inhibition efficiency and surface coverage of aqueous extract of HRF on MS in SOWW at room temperature (303K)

Tabela 1. Brzina korozije, efikasnost inhibicije i pokrivenost površine vodenog ekstrakta HRF na MS u SOWW na sobnoj temperaturi (303K)

Inhibitor (HRF % v/v)	Corrosion rate (CR) mdd	IE%	Surface coverage (θ)
0	15.55	-	-
2	6.53	58	0.580
4	5.44	65	0.650
6	4.35	72	0.720
8	3.11	80	0.800
10	2.80	82	0.820

The increase in the concentration increases the surface coverage due to adsorption which increases the active sites on the metal surface. Hence, the inhibition efficiency increases with increase in the concentration. The presence of lone pair of electrons on the hetero atoms of oxygen facilitates the formation of co-ordinate bonds with the metal and is responsible for corrosion inhibition. Most of the effective organic inhibitors used in petroleum industry have hetero atom such as O, N and S along with multiple bonds in their molecules through which they are adsorbed on the metal surface [20, 21].

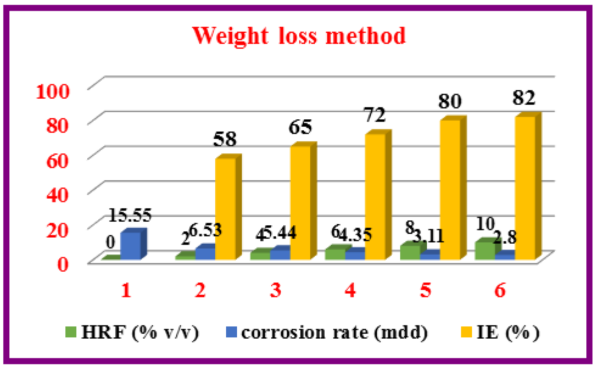

Figure 2. Column chart: HRF (% v/v), corrosion rate (mdd) and IE (%) of MS in SOWW medium in absence and presence of different concentrations of inhibitor (HRF)

Slika 2. Kolona grafikon: HRF (% v/v), brzina korozije (mdd) i IE (%) MS u SOWW medijumu u odsustvu i prisustvu različitih koncentracija inhibitora (HRF)

Adsorption isotherm

An adsorption isotherm gives the direct relationship between the corrosion inhibition efficiency with the degree of surface coverage at constant temperature for different concentrations of inhibitor solutions. The adsorption isotherm provides the basic information about the nature of interaction between the mild steel surface and inhibitor molecular constituents [22]. Adsorption of the corrosion inhibitor molecules occurs on the mild steel surface by the displacement of molecule of water adsorbed on the metal surface. Also, the adsorption depends on the temperature, chemical composition and constituents of inhibitor and the electrochemical potential at the metal-solution interface [23, 24].

Langmuir adsorption isotherm was proposed to account for the adsorption of the corrosion inhibitor molecules on the surface of metal. From the isotherm, the linear relationship between θ and concentration of inhibitor can be found.

Advantage of use of Langmuir adsorption isotherm

The Langmuir isotherm model is one of the most common and simple to use isotherms due to its effectiveness in low concentrations, exibility with computer simulations, and easy handling. The Langmuir adsorption isotherms predict linear adsorption at low adsorption densities and a maximum surface coverage at higher solute metal concentrations.

To specify the adsorption mode of *Hibiscus rosa-sinensis* flower extract molecules on the mild steel surface in the SOWW medium, the degree of surface coverage (θ) for different concentrations of inhibitor (2, 4, 6, 8 and 10 % v/v) at room temperature was found from the weight loss method. According to Langmuir adsorption isotherm, the following equation relates the surface coverage (θ) and inhibitor concentration, C.

$$C/\theta = 1/K_{ads} + C$$

The plot of Langmuir adsorption isotherm, C/θ vs C is shown in Figure 3. The ΔG^0_{ads} is calculated from the equation.

$$\Delta G^0_{ads} = -2.303\ RT \log (K_{ads} \times 55.55)$$

The values of adsorption parameters obtained from Langmuir adsorption includes free energy of adsorption (ΔG^0_{ads}), equilibrium constant (K_{ads}), coefficient of correlation (R^2), slope and intercept values are presented in Table 2. The R^2 value approaches unity. The value of standard free energy of adsorption is -8.905 kJmol^{-1} indicates the physical adsorption process. The negative sign indicates that the adsorption of the inhibitor (HRF) constituents on the metal is a spontaneous process and adsorption takes place very easily without requiring any extra energy. These observations indicates that the adsorption of inhibitor molecules on the metal surface obey the Langmuir adsorption isotherm [25, 26].

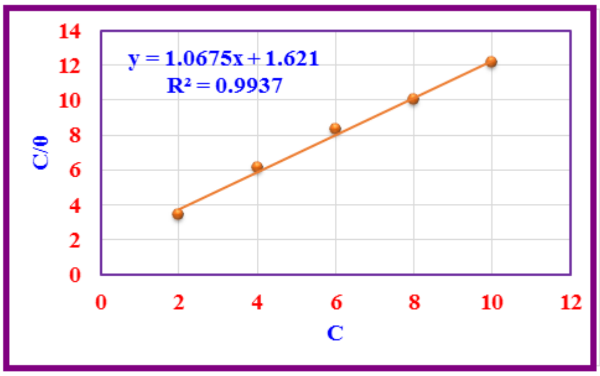

Figure 3. Langmuir adsorption isotherm for the inhibition effect of aqueous extract of HRF on mild steel corrosion in SOWW at room temperature (303 K)

Slika 3. Langmuir-ova adsorpciona izoterma za inhibicioni efekat vodenog ekstrakta HRF na blagu koroziju čelika u SOWW na sobnoj temperaturi (303 K)

Table 2. Adsorption parameters obtained from Langmuir adsorption isotherm for the corrosion inhibitive effect of aqueous extract of HRF on mild steel in SOWW

Tabela 2. Parametri adsorpcije dobijeni iz Langmuir-ove adsorpcione izoterme za inhibicijski efekat vodenog ekstrakta HRF na meki čelik u SOWW

R^2	Slope	Intercept	K_{ads}	ΔG^0_{ads}
0.9937	1.0675	1.621	0.617	-8.905

Electrochemical study

Analysis of polarization study

Polarization study is employed to confirm the formation of protective layer on the MS surface. If a protective layer is formed, the linear polarization resistance (LPR) value increases and the corrosion current (I_{corr}) value decreases. The polarization curves of MS immersed in different environments are shown in Figure 4 and 5.

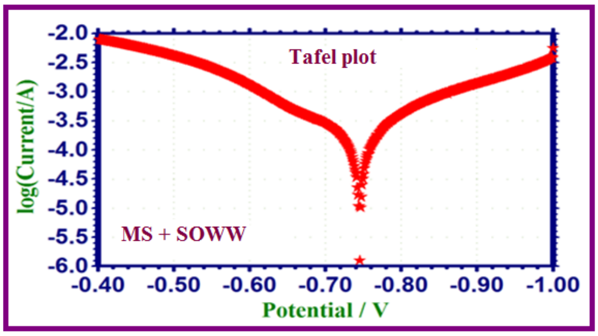

Figure 4. Polarization curves for corrosion of MS immersed in SOWW

Slika 4. Polarizacione krive za koroziju MS uronjenog u SOWW

Polarization parameters such as corrosion potential (E_{corr}) value, corrosion current (I_{corr}) value, linear polarization resistance (LPR) value and Tafel slopes (b_c = cathodic slope and b_a = anodic slope) are calculated from the polarization curves. The values are summarized in Table 3. It is noticed from the Table 3 that when mild steel is immersed in SOWW medium, the corrosion potential (E_{corr}) value is -745 mV vs SCE. The corrosion current (I_{corr}) value is 3.14 x 10^{-4} A/cm^2. The linear polarization resistance (LPR) value is 127 ohm cm^2.

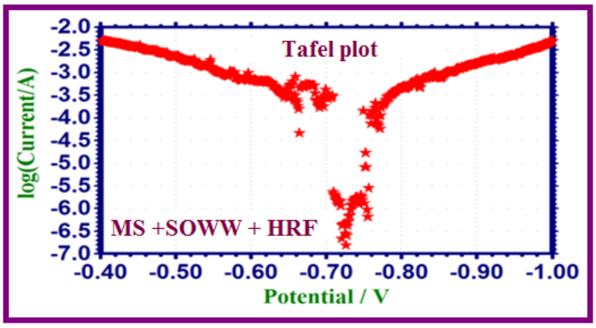

Figure 5. Polarization curve for corrosion of MS immersed in SOWW + 10 % v/v HRF

Slika 5. Kriva polarizacije za koroziju MS uronjenog u SOWW + 10 % v/v HRF

The addition of 10 % v/v of inhibitor (HRF) in SOWW medium the corrosion potential (E_{corr}) value is shifted from -745 to -726 mV vs SCE. This shift is very small. The displacement of E_{corr} is < 85 mV, hence the inhibitor can be classified as a mixed type inhibitor [27] controlling both anodic reaction and cathodic reaction by forming a protectivefilm [28, 29]. When 10 % v/v of HRF is added to the SOWW medium the linear polarization resistance (LPR) value increases from 127 to 662 ohm cm^2. The corrosion current (I_{corr}) value decreases from 3.14 x 10^{-4} to 4.93 x 10^{-6} A/cm^2. The results confirm that a protective layer is formed on the MS surface. Hence the corrosion of MS is controlled.

Table 3. Potentiodynamic polarization parameters of mild steel in SOWW in the absence and presence of aqueous extract of inhibitor (HRF)

Tabela 3. Potenciodinamički parametri polarizacije mekog čelika u SOWW u odsustvu i prisustvu vodenog ekstrakta inhibitora (HRF)

Concentration of aqueous extract of CHL (% v/v)	E_{corr} mV vs SCE	Tafel slope mV/decade		LPR Ohm cm^2	I_{corr} A/cm^2
		b_c	b_a		
0	-745	183	183	127	3.14 × 10^{-4}
10	-726	141	160	662	4.93 × 10^{-6}

Analysis of AC impedance spectra

AC impedance spectra provide information about the formation of protective layer on the MS. If a protective layer is formed on the MS surface, the charge transfer resistance (R_t) value increases; double layer capacitance (C_{dl}) value decreases and the impedance [log (z/ohm)] value enhances. The AC impedance spectra of MS immersed in SOWW medium in presence of inhibitor HRF are shown in Figure 6 and 7. The Nyquist plot and the Bodeplots are shown in Figure 6 and 7 respectively. The corrosion parameters, namely the charge transfer resistance (R_t), double layer capacitance value (C_{dl}) and impedance [log(z/ohm)] value are summarized in Table 4.

It is noticed from the Table 4 that when MS is immersed in SOWW, the charge transfer resistance (R_t) value is 17.435 ohm cm^2. The double layer capacitance (C_{dl}) value is 2.925 x 10^{-7} F/cm^2. The impedance [log (z/ohm)] value is 1.391 and the phase angle value is 26.58°. In the presence of inhibitor HRF, the charge transfer resistance (R_t) value increases from 17.435 ohm cm^2 to 22.948 ohm cm^2. The double layer capacitance (C_{dl}) value decreases from 2.925x10^{-7} F/cm^2 to 2.223x10^{-7} F/cm^2. The impedance [log (z/ohm)] value increases from 1.391 to 1.936. The phase angle value increases from 26.58° to 28.95°. The results conclude that a protective layer is formed on the MS surface [11, 31, 32]. It restricts the flow of electrons from the MS to the medium. Hence corrosion of MS is controlled.

Table 4. Electrochemical impedance parameters of mild steel in SOWW in the absence and presence of aqueous extract of inhibitor (HRF)

Tabela 4. Parametri elektrohemijske impedanse mekog čelika u SOWW u odsustvu i prisustvu vodenog ekstrakta inhibitora (HRF)

Concentration of aqueous extract of HRF, (% v/v)	Nyquist plot		Bode plot	
	R_t, Ohm cm^2	C_{dl}, F/cm^2	Impedance [log(Z/Ohm)]	Phase angle, (°)
0	17.435	2.925 × 10^{-7}	1.391	26.58
10	22.948	2.223 × 10^{-7}	1.936	28.95

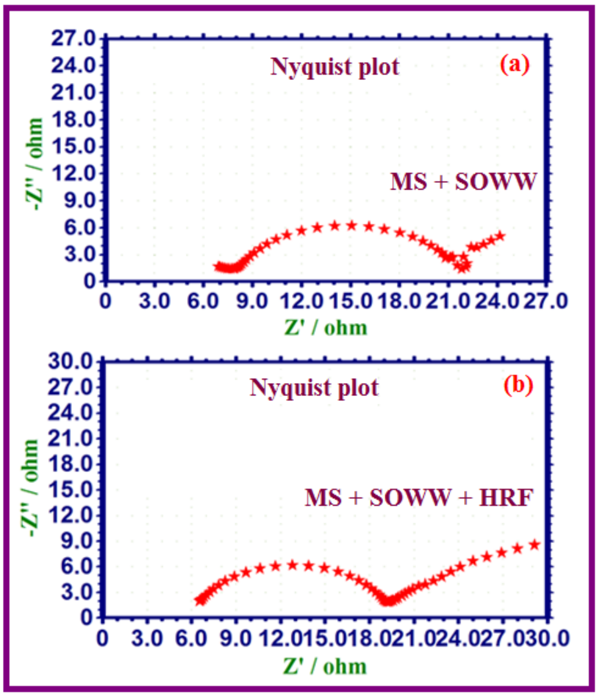

Figure 6. Nyquist plot of mild steel in different environments (a) SOWW without inhibitor (b) with 10 % v/v aqueous extract of inhibitor (HRF)

Slika 6. Nyquist-ov dijagram mekog čelika u različitim okruženjima (a) SOWW bez inhibitora (b) sa 10% v/v vodenog ekstrakta inhibitora (HRF)

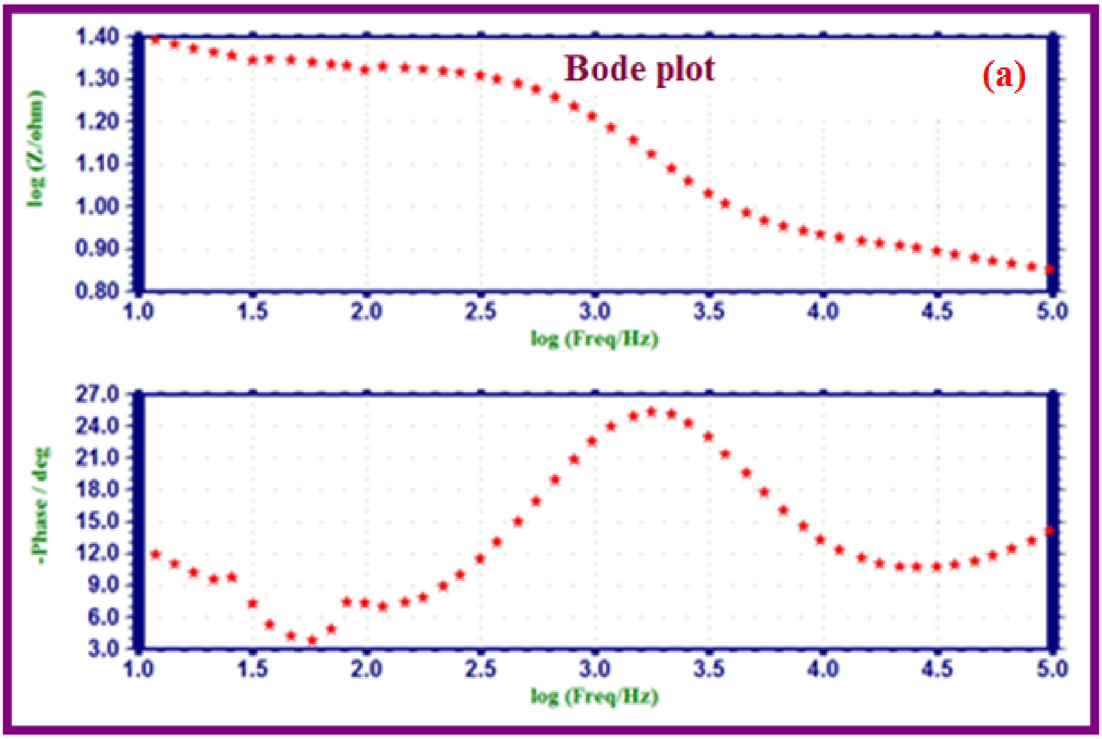

Figure 7a. Bode plots of mild steel in SOWW without inhibitor (blank)

Slika 7a. Bode-ovi dijagrami mekog čelika u SOWW bez inhibitora (prazno)

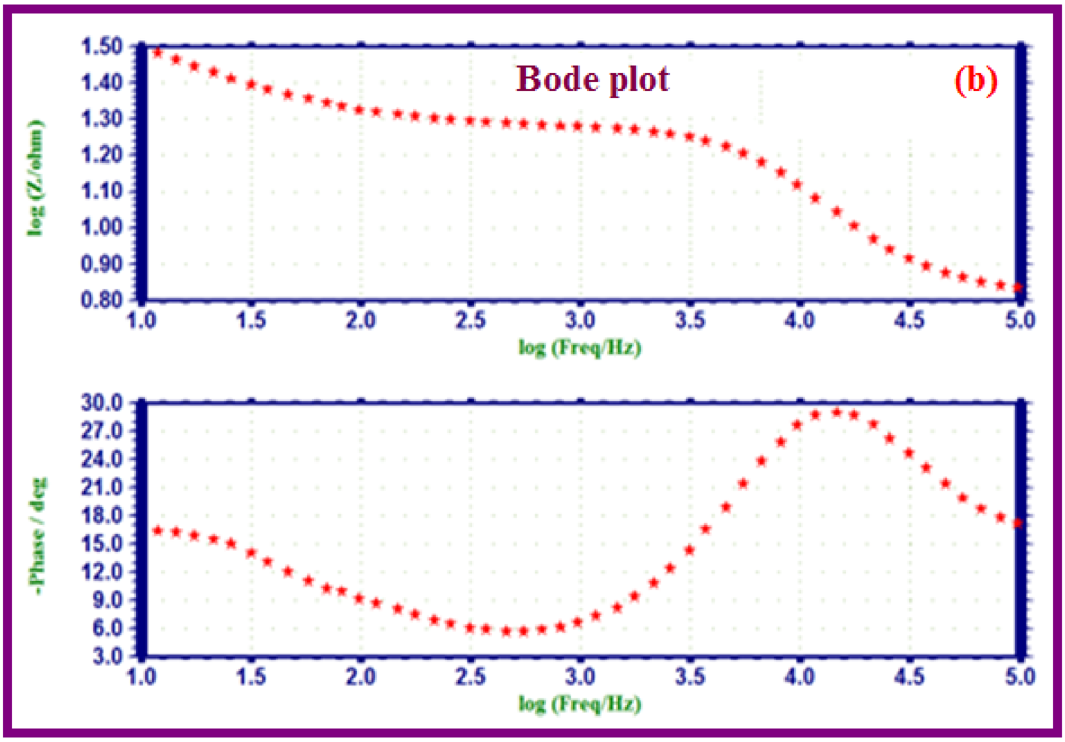

Figure 7b. Bode plots of mild steel in SOWW with 10 % v/v aqueous extract of inhibitor (HRF)

Slika 7b. Bode-ovi dijagrami mekog čelika u SOWW sa 10% v/v vodenog ekstrakta inhibitora (HRF)

Analysis of FTIR spectra

FTIR spectra have been used to analyse the protective film formed on the metal surface. A few drops of an aqueous extract of *Hibiscus rosa-sinensis* flower were dried on a glass plate. A solid mass was obtained. It was blended with KBr and converted into pellet. Its FTIR spectral pattern is recorded and given in Figure 8, the assignments are shown in Table 5. A broad band appears at 3423.09 cm^{-1} is attributed to -OH group. The band observed at 2924.60 cm^{-1} and 2855.20 cm^{-1} represents the aliphatic C-H stretching. The peak at 1637.75 cm^{-1} shows the presence of C=O (aromatic ketone) and aromatic ring (C=C) group. The peak at 1385.94 cm^{-1} has been assigned to CH bending frequency. The band 1244.85 cm^{-1} indicates the presence of C-O stretching group. The peak at 1069.75 cm^{-1} is assigned to C-O-C stretching vibration. The C-H out of plane bending (oop) absorbs at 899.80 cm^{-1} and 668.44 cm^{-1}.

The FTIR spectrum of the protective layer formed on the mild steel surface after immersion in the solution containing SOWW with 10 % v/v of inhibitor (HRF) solution is shown in Figure 9. The respective absorption frequencies are given in Table 5. The shift at 3423.09 cm^{-1} to 3382.19 cm^{-1} can be attributed to the presence of -OH stretching. The slight shift from 2924.60 cm^{-1} to 2925.10 cm^{-1} indicates the presence of C-H bond. The peak has shifted from 1637.75 cm^{-1} to 1629.76 cm^{-1} indicate the presence of C=O and C=C aromatic ring group.

Table 5. FTIR spectral data for the aqueous extract of HRF and the scratched film from mild steel surface after immersion in SOWW with 10 % v/v HRF

Tabela 5. FTIR spektralni podaci za vodeni ekstrakt HRF i izgrebani film sa površine mekog čelika nakon potapanja u SOWW sa 10 % v/v HRF

Stretching frequency, cm^{-1}		Various functional groups
Dried aqueous extract of HRF	Protective film formed on mild steel surface	
3423.09	3382.19	-OH stretching
2924.60	2925.10	-CH stretching
2855.20	-	-CH stretching
1637.75	1629.76	C=O/C=C stretching
1385.94	1454.39	-CH bending
1244.85	-	C-O stretching
1069.75	1022.37	C-O-C stretching
899.80	872.82	CH “oop”
668.44	688.46	CH “oop”
-	469.70	γ-Fe_2O_3

The C-H bending frequency has shifted from 1385.94 cm^{-1} to 1454.39 cm^{-1}. The frequency 1244.85 cm^{-1} for C-O group is disappeared. The C-O-C stretching frequency has shifted from 1069.75 cm^{-1} to 1022.37 cm^{-1}. The peak C-H "oop" bending shifted from 899.80 cm^{-1} to 872.82 cm^{-1} and 618.11 cm^{-1} to 694.82 cm^{-1}. The new absorption band at 469.70 cm^{-1} probably originates from the Fe^{2+}-*Hibiscus rosa-sinensis* flower complex formation. This shows that due to interaction between the metal and the active constituents present in the flower extract, there is a change in the chemical nature of the active constituents [33, 34].

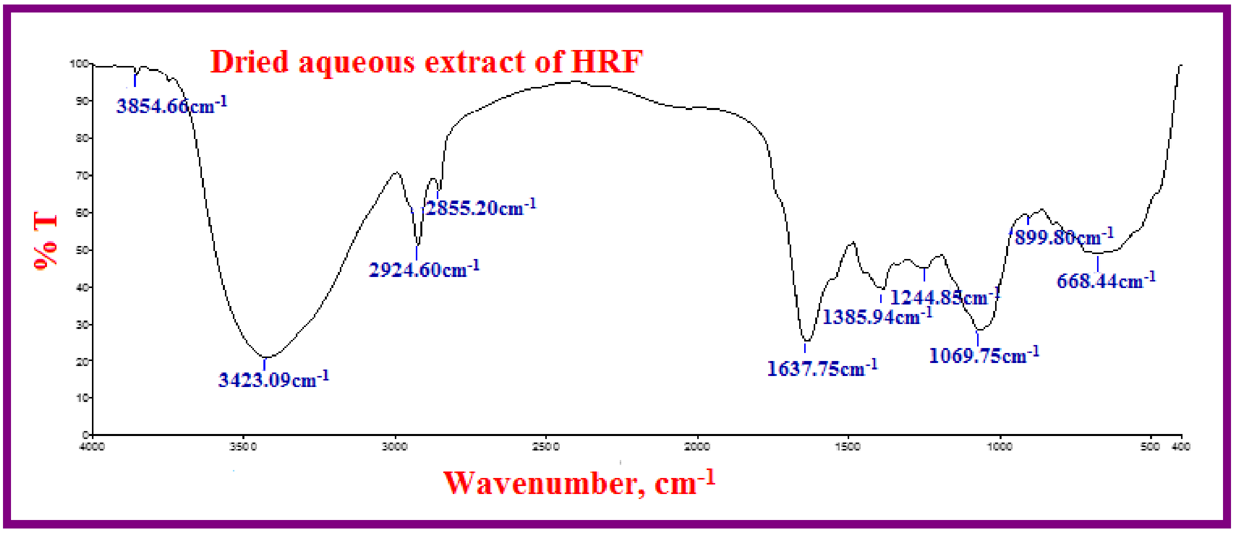

Figure 8. FTIR spectrum of dried aqueous extract of pure inhibitor (HRF)

Slika 8. FTIR spektar osušenog vodenog ekstrakta čistog inhibitora (HRF)

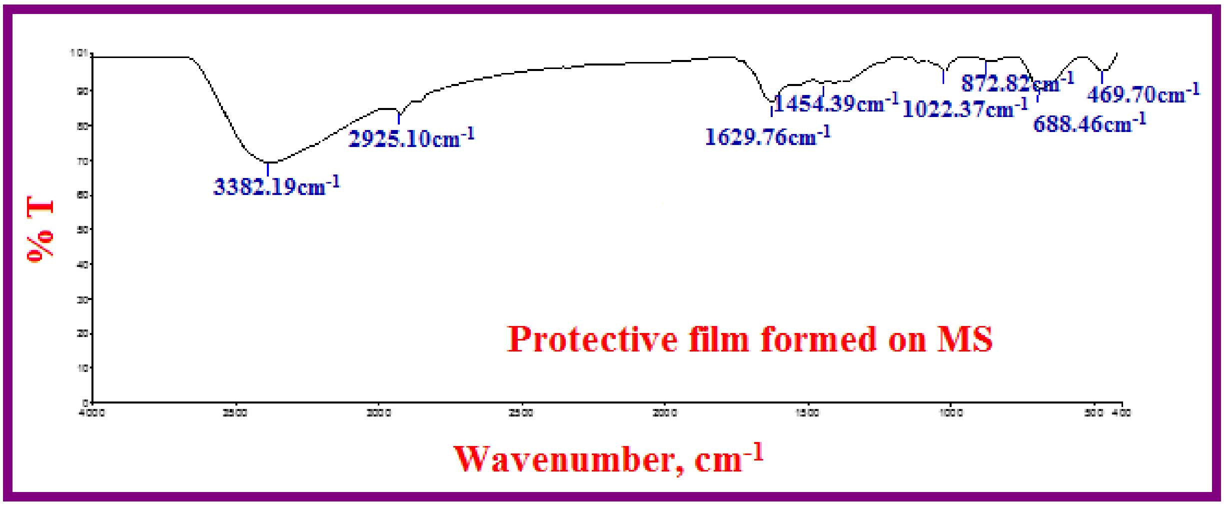

Figure 9. FTIR spectrum of scratched film from the mild steel surface after immersion in SOWW with 10 % v/v aqueous extract of inhibitor (HRF)

Slika 9. FTIR spektar izgrebanog filma sa površine mekog čelika nakon potapanja u SOWW sa 10% v/v vodenog ekstrakta inhibitora (HRF)

Analysis of SEM studies

SEM image is employed in the analysis of surface morphology of the specimen. The SEM images of MS in various environments are shown in Figure 10. The SEM image of polished MS is given in Figure 10a. The SEM image of polished MS immersed in SOWW is shown in Figure 10b. The SEM image of the MS immersed in SOWW in presence of inhibitor HRF are given in Figure 10c. The SEM images of polished MS are appeared to be smooth. The SEM image of the MS in SOWW environment is seemed to be rough. The noticeable cavities are present on the MS surface. The SEM image of the MS in SOWW and inhibitor (HRF) are appeared to be significantly smooth than in the SOWW medium. Thus the SEM study reveals the MS surface is protected by the formation of non-porous smooth thin film in the presence of inhibitor (HRF)[35].

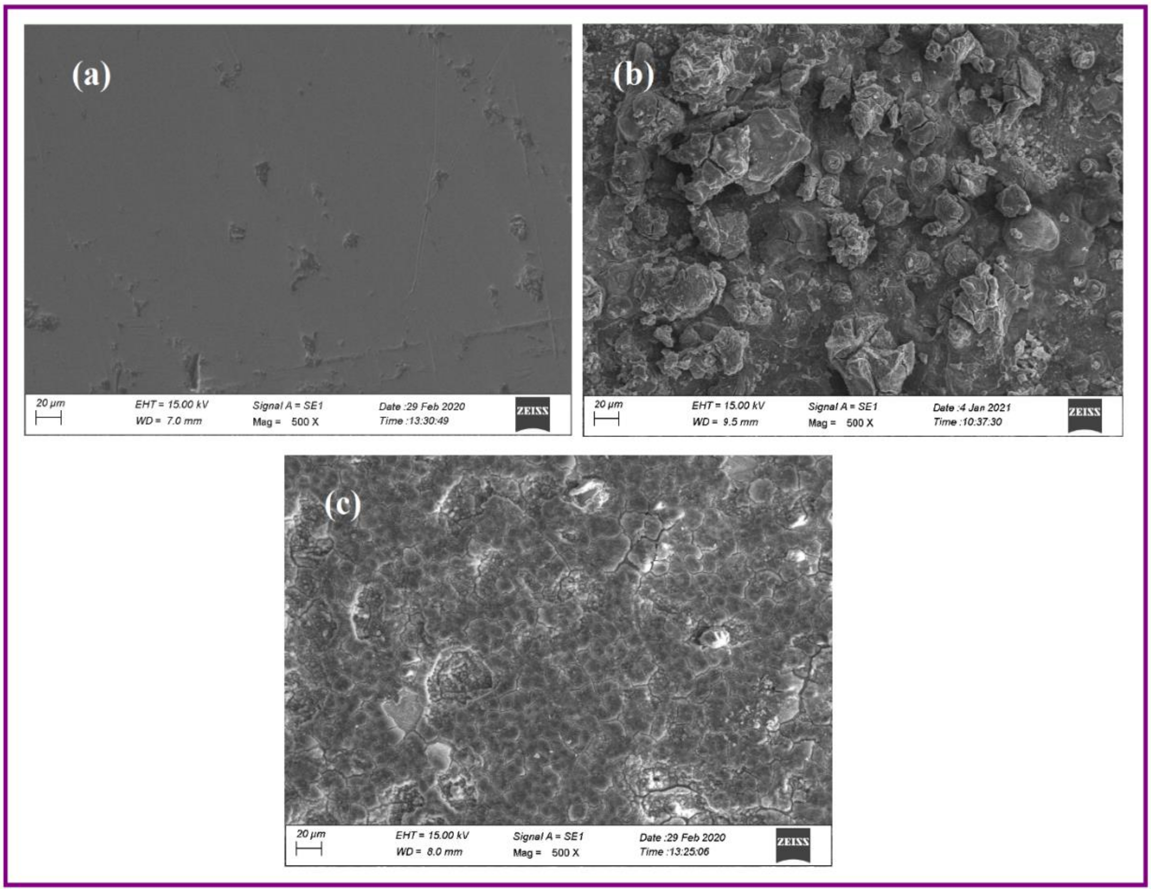

Figure 10. SEM micrographs of (a) Polished MS (b) MS immersed in SOWW (c) MS immersed in SOWW with 10 % v/v aqueous extract of HRF

Slika 10. SEM mikrografije (a) Poliranog MS (b) MS uronjenog u SOWW (c) MS uronjenog u SOWW sa 10% v/v vodenog ekstrakta HRF

Mechanism of corrosion inhibition

The extract of the flower contains many chemical constituents such as cyanidin, quercetin, hentriacontane, calcium oxalate, thiamine, riboflavin, niacin and ascorbic acidsThey adsorbed on the metal surface and form protective film on the metal surface and thus corrosion is controlled [36].

4. CONCLUSION

The following conclusions can be obtained from the results.

- The weight loss method reveals that 82 % inhibition efficiency is observed in controlling corrosion of mild steel in simulated oil well water containing corrosion of an aqueous extract of *Hibiscus rosa-sinensis* flower.
- Potentiodynamic polarization study shows that mixed type of inhibitor.
- AC impedance spectra reveal that protective film is formed on the metal surface.
- The presence of active principle component in the aqueous extract of HRF has been confirmed by FTIR spectra.
- The smoothness of the metal surface has been studied by SEM.

5. REFERENCE

[1] K.Tamalmani, H.Husin (2020) Review on Corrosion Inhibitors for Oil and GasCorrosion Issues, Applied Sciences, 10(10), 3389.

[2] L.T.Popoola, A.S.Grema, G.K.Latinwo, B.Gutti, A.S.Balogun (2013) Corrosion problems during oil and gas production and its mitigation, International Journal of Industrial Chemistry, 4(35), 1-15.

[3] M.N.Rahuma, B.Kannan (2014) Corrosion in Oil and Gas Industry: A Perspective on Corrosion Inhibitors, J. of Material Sciences and Engineering, 3(3), 110-119.

[4] L.N.Emembolu, O.D.Onukwuli, C.J.Umembamalu, C.O.Aniagor (2020) Evaluation of the corrosion inhibitory effect of NapoleonaeaImperalis leaf extract on mild steel in a 1.3 M H_2SO_4, medium, J. of Bio- and Tribo-Corrosion, 6(128), 1-15.

[5] I.Y.Suleiman, A.Kasim, S.R.Ochu (2019) Anti-corrosion Properties of Ethanol Extract of Cardiospermumhalicacabum Leaf on Steel Pipelines in Acidic Environment, Journal of Bio- and Tribo-Corrosion, 5(65), 1-13.

[6] K.Kavitha, H.Benitasherine, S.Rajendran (2021) Cardiospermum halicacabum leaves extract as a green corrosion inhibitor for mild steel in simulated oil well water medium, International Journal of Corrosion and Scale Inhibition, 10(4), 1646–1660.

[7] A.S.Fouda, A.S.Abousalem, G.Y.EL-Ewady (2017) Mitigation of corrosion of carbon steel in acidic solutions using an aqueous extract of Tiliacordata as green corrosion inhibitor, International Journal of Industrial Chemistry, 8, 61–73.

[8] R.Kusumastuti, R.I.Pramana, J.W.Soedarsono (2017)The Use of MorindaCitrifolia as a Green Corrosion Inhibitor for Low Carbon Steel in 3.5% NaCl Solution, International Conference on Chemistry, Chemical Process and Engineering, 020012-1–020012-9.

[9] G.I.Udom, G.A.Cookey, A.Abia (2017) The Effect of Acanthus montanus Leaves Extract on Corrosion of Aluminium in Hydrochloric Acid Medium, Current Journal of Applied Science and Technology, 25(2), 1-11.

[10] A.El-A.S.Fouda, A.A.El-Hossiany, H.M.Ramadan (2017) Calotropisprocera plant extract as green corrosion inhibitor for 304 stainless steel in hydrochloric acid solution, Zastita Materijala, 58(4), 541 - 555.

[11] K.Anuradha,R.Vimala,B.Narayanasamy, J.A.Selvi, S.Rajendran (2008) Corrosion Inhibition of Carbon Steel in Low Chloride Media by an Aqueous Extract of Hibiscus rosa-sinensis Linn, Chemical Engineering Communications, 195, 352–366.

[12] S.Rajendran, J.Jeyasundari, P.Usha, J.A.Selvi, B.Narayanasamy, A.P.P.Regis, P.Rengan (2009) Corrosion Behaviour of Aluminium in the Presence of an Aqueous Extract of Hibiscus Rosa-sinensis, Portug.Electrochim.Acta, 27(2), 153-164.

[13] P.S.Desai (2015) Hibiscus rosa-sinensis (JASUD) leaves extracts used as corrosion inhibitors for mild steel in hydrochloric acid, European Journal of Pharmaceutical and Medical Research, 2(1),470-485.

[14] M.Goutam, D.K.Prabir,D.Pranjal, S.Akhsay, K. Shymodip, S.Pintu, M.Debasmita, S.Argha, N. Tarique, K.Chandan, R.C.Sangita (2018) Authentication and Photochemical Screening of Hibiscus Rosa Sinensis, International Journal of Research and Analytical Reviews, 5(4), 712u-718u.

[15] I.M.Khan, R.Rahman, A.Mushtaq,M.Rezgui (2017) Hibiscus rosa-sinensis L. (Malvaceae): Distribution, chemistry and uses, International Journal of Chemical and Biochemical Sciences, 12, 147-151.

[16] R.Geethanjali, S.Subhashini (2015) Investigation of Corrosion Inhibition Efficiency of Some Synthesized Water Soluble Terpolymers on N-80 Steel in HCl, NaCl and Simulated Oil Well Water, Portug. Electrochim. Acta, 33(2), 85-104.

[17] G.Wranglen (1986) Introduction to Corrosion and Protection of Metals, London: Chapman and Hall, p.236.

[18] S.S.Sawant, D.Khandeparker, A.Tulaskar, K. Venkat,A.Garg (1995) Corrosion and microfouling of copper and its alloys in a tropical marine waters of India (Mangalore), Indian Journal of Chemical Technollogy, 2, 322-326.

[19] K.Kavitha, H.B.Sherine, S.Rajendran (2021) Anti-corrosive properties of an aqueous extract of Chrysanthemum indicum flower, International Journal of Corrosion and Scale Inhibition, 10(2), 783–800.

[20] S.Muralidharan,S.V.Iyer (1997) The influence of N-heterocycles on corrosion inhibition and hydrogen permeation through mild steel in acidic solutions, Anti-Corrosion Methods and Materials, 44, 100.

[21] R. Saratha, S.V. Priya, P. Thilagavathy (2009) Investigation of Citrus aurantiifolia leaves extract as corrosion inhibitor for mild steel in 1 M HCl, E-Journal of Chemistry, 6(3), 785-795.

[22] E.A.Noor, A.H.Al-Moubaraki (2008) Thermodynamic study of metal corrosion and inhibitor adsorption processes in mild steel/1-methyl-4 [4'(-X)-styrylpyridinium iodides/hydrochloric acid systems, Materials Chemistry and Physics, 110, 145-154.

[23] H.Shao, J.Wang, Z.Zhang, J.Zhang, C.Cao (2003) The cooperative effect of calcium ions and tartrate ions on the corrosion inhibition of pure aluminum in an alkaline solution, Materials Chemistry and Physics, 77, 305-309.

[24] A.A.S.Begum, R.M.A.Vahith, V.Kotra, M.R.Shaik, A.Abdelgawad, E.M.Awwad, M.Khan (2021) Spilanthesacmella leaves extract for corrosion inhibition in acid medium, Coatings, 11(106), 1-23.

[25] O.F.Nwosu, E.Osarolube, L.A.Nnanna, C.S.Akoma, T.Chigbu (2014) Acidic corrosion inhibition of Piper guineense seed extract on Al alloy, American Journal of Materials Science, 4(4), 178-183.

[26] T.Sathiyapriya,G.Rathika (2019) Corrosion inhibition efficiency of human black hair extract on mild steel in 1M H_2SO_4 media, Indian Journal of Chemical Technology, 26, 216-223.

[27] H.M.A.El-Lateef, M.A.Abo-Riya,A.H.Tantawy (2016) Empirical and quantum chemical studies on the corrosion inhibition performance of some novel synthesized cationic gemini surfactants on carbon steel pipelines in acid pickling processes, Corrosion Science, 108, 94-110.

[28] M.Lebrini, F.Robert, H.Vezin, C.Roos (2010) Electrochemical and quantum chemical studies of some indole derivatives as corrosion inhibitors for C38 steel in molar hydrochloric acid, Corrosion Science, 52, 3367-3376.

[29] F.El-T.Heakal, M.A.Deyab, M.M.Osman, M.I. Nessim, A.E.Elkholy (2017) Synthesis and

assessment of new cationic Gemini surfactants as inhibitors for carbon steel corrosion in oilfield water, Journal of RSC Advances, 7, 47335-47352.

[30] A.SuriyaPrabha, K.Kavitha, H.BenitaSherine, S. Rajendran (2020) Inhibition of corrosion of mild steel in simulated oil well water by an aqueous extract of Andrographispaniculata, Indian Journal of chemical Technology, 27, 452-460.

[31] J.J.M.Praveena, J.A.Clara, S.S.Rajendran, A.J. Amalraj (2021) Inhibition of corrosion of mild steel in well water by an aqueous extract of soapnut (SapindusTrifoliatus), Zastita Materijala, 62(4), 277–290.

[32] P.Deivanayagam,S.Selvaraj (2018) Cardiospermum halicababum leaves extract as green inhibitor for corrosion of brass in 1.0N hydrochloric acid solution, International Journal for Research in Engineering Application and Management, 4(6), 292-298.

[33] M.A.Deyab, M.M.Osman, A.E.Elkholy,F.El-T.Heakal (2017) Green approach towards corrosion inhibition of carbon steel in produced oilfield water using lemongrass extract, Journal of RSC Advances, 7, 45241-45251.

[34] R.M.Silverstein, F.X.Webster, D.J.Kiemle (2005) Spectroscopic identification of organic compounds, John Wiley and Sons, seventh edition, 82, 106-115.

[35] I.Y.Suleiman, A.M.Sani, O.O.Clifford (2020) Characterization and corrosion behaviours of AISI 316 in hydrochloric environment at various concentrations, Zastita Materijala, 61(3), 220–228.

[36] P.Ruban, K.Gajalakshmi (2012) In vitro antibacterial activity of Hibiscus rosa-sinensis flower extract against human pathogens. Asian Pac J Trop Biomed. 2(5), 399-403. doi:10.1016/S2221-1691(12)60064-1.

IZVOD

INHIBICIJA KOROZIJE MEKOG ČELIKA U SIMULIRANOJ VODI IZ BUNARA VODENIM EKSTRAKTOM CVETA *Hibiscus rosa-sinensis*

Vodeni ekstrakt cveta Hibiscus rosa-sinensis (HRF) korišćen je kao inhibitor korozije u kontroli korozije mekog čelika u simuliranoj vodi iz bunara (SOWW). Metoda gubitka težine otkriva da 10% v/v ekstrakta nudi 82% efikasnost inhibicije (IE) za SOWW uronjen u meki čelik (MS). Mehanistički aspekti inhibicije korozije su istraženi proučavanjem polarizacije i spektra impedanse naizmenične struje. Studija polarizacije otkriva da je mešoviti tip inhibitora u prisustvu inhibitornog sistema. Efekat inhibicije korozije je potvrđen povećanjem vrednosti otpora linearne polarizacije i smanjenjem vrednosti struje korozije. Zaštitni sloj koji se formira na površini metala potvrđuju spektri impedanse naizmenične struje. Ovo potvrđuje činjenica da dolazi do povećanja vrednosti otpora prenosa naelektrisanja i smanjenja vrednosti kapacitivnosti dvostrukog sloja. Adsorpcija molekula inhibitora je podređena Langmuir-ovoj adsorpcionoj izotermi. Zaštitni premaz karakteriše FTIR spektroskopija. To potvrđuje da je inhibitor koordinirao sa jonima gvožđa na površini metala preko aktivne komponente b-sitosterola, kvercetina i kempferola-3-ksilozilglukozida. Morfologija površine je ispitana skenirajućim elektronskim mikroskopom (SEM). Dakle, vodeni ekstrakt cveta Hibiscus rosa-sinensis sa simuliranom vodom iz bunara ima dobar korozivni inhibitivni efekat na cevovode od mekog čelika.

Ključne reči: *Hibiscus rosa-sinensis, simulirana naftna bušotina, meki čelik, inhibicija korozije, morfologija površine, SEM.*

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