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Rosa damascena (Damask Rose) as corrosion inhibitor for mild steel in simulated oil well water medium

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Abstract

The mild steel pipeline system in the oil and gas industry is the heart for transportation of crude and refined petroleum. However, continuous exposure of the pipeline surfaces to impurities and sources of corrosion such as sulfur and chromate is totally unavoidable. Vast employment of commercial corrosion inhibitors to minimize the corrosion is being restrained due to toxicity towards the environment. The emergence of "green" chemistry has led to the use of plant extracts which have proven to be good corrosion inhibitors. This paper aims to provide insight into carrying out further investigation under this research theme for accurate inhibition efficiency measurement. The impact of an aqueous extract of *Rosa damascena* flower (RDF) in controlling the corrosion of mild steel in simulated oil well water (SOWW) medium has been demonstrated by weight loss method and electrochemical measurements. The surface morphology is examined with the help of scanning electron microscopy (SEM) and the surface roughness analysis is done by atomic force microscopy (AFM). Weight loss method reveals that 10% v/v of the extract offers a maximum corrosion inhibition efficiency of 96%. The potentiodynamic polarization method was used to study the mechanistic aspects of corrosion inhibition. Corrosion potential values are shifted. Linear polarization resistance value increases and corrosion current value decreases with increase in concentration of RDF. The potentiodynamic polarization study reveals that RDF acts as mixed type inhibitor with primarily anodic effectiveness. SEM and AFM documented the development of shielding coating on the mild steel surface. The results obtained show that *Rosa damascena* flower extract could serve as an excellent green corrosion inhibitor.

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Keywords: *Rosa damascena flower, corrosion inhibitor, mild steel, simulated oil well water*.

Introduction

Pipelines play an extremely important role throughout the world as a means of transporting gases and liquids over long distances from their sources to the ultimate consumers [1]. Corrosion of mild steel in the presence of oilfield water is a corrosion problem across petroleum industries [2–4]. Simulated oil well water exists in natural gas oil reservoirs and it is rich in huge amounts of dissolved salts such as chloride and sulfate besides corrosive dissolved gases like CO_2 and H_2S [5]. Natural products, which are available in nature and can be extended, were a good alternative for corrosion inhibitor. Several studies have developed a corrosion inhibitor material, which is derived from plants such as *Catharanthus roseus* [6], *Acanthus montanus* leaves [7], *Plantago* [8], *Acalypha indica* [9], Watermelon rind extract [10], Linseed oil [11], *Thymus algeriensis* [12], *Cryptocarya nigra* [13] and *Aganonerion polymorphum* [14].

Rajan Anitha *et al*. studied the *Rosa damascena* leaves extract as a green corrosion inhibitor on reinforced steel rebar [15]. Xia Wang *et al*. discussed the rose, gardenia and *Solanum violaceum* corrosion inhibitor effect of mild steel in 1 M HCl solution [16]. Aisha M. Al-Turkustani studied the corrosion inhibition of steel in sulphuric acid by aqueous extract of Rose and Rose water [17]. However, in the present study we have investigated the corrosion inhibitive properties of the aqueous extract of *Rosa damascena* flower (Damask Rose) (Figure 1) in simulated oil well water solution using the weight loss method and electrochemical polarization method. The protective film was analysed by SEM and AFM studies.

Figure 1. *Rosa damascena* flower.

Experimental

Preparation of inhibitor

The aqueous extract of *Rosa damascena* flower (RDF) were prepared by the method of soxhlet extraction. About 100 g of powdered plant of *Rosa damascena* 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 [18].

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 $1.0\times4.0\times0.2$ cm^3 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 *Rosa damascena* 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 [19]. The corrosion rates were calculated using the following equation [20].

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

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:

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

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, the corrosion resistance of MS immersed in various test solutions was measured by polarization study. All the experiments were done at room temperature.

Polarization study

Polarization studies were carried out in a CHI electrochemical work station with impedance model 660A. It was provided with an i_R 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 the 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.

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 CARL ZEISS make model EVO-18.

Atomic force microscopy (AFM)

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 atomic force microscopy (AFM) using SPM Veeco diInnova connected with the software version V7.00 and the scan rate of 0.7 Hz.

Results and Discussion

Analysis of results of the weight loss method

Inhibition efficiency of an aqueous extract of *Rosa damascena* flower (RDF) in controlling corrosion of mild steel immersed in simulated oil well water in absence and presence of inhibitor for a period of one day are given in Table 1. It is observed that RDF extract has good inhibition efficiency. As the concentration of RDF increases inhibition efficiency also increases. Aqueous extract of RDF offers 96% inhibition efficiency. So corrosive agents and water molecules cannot stick on the metal surface. Hence inhibition efficiency increases [21].

Table 1. The corrosion rate and the inhibition efficiency of MS in SOWW medium in different concentrations of inhibitor (RDF).

Concentration of inhibitor (RDF), ppm	Corrosion rate (*CR*), mdd	*IE*%
0	14.55	–
2000	3.64	75
4000	2.76	81
6000	1.75	88
8000	1.31	91
10000	0.58	96

Analysis of polarization study

Potentiodynamic polarization measurements can provide important information about the kinetic of cathodic and anodic corrosion reactions [22]. Electrochemical corrosion parameters involve the corrosion potential (E_{corr}), corrosion current density (I_{corr}), as well as the anodic and cathodic Tafel constants (b_a and b_c) respectively. The potentiodynamic polarization curves of mild steel immersed in SOWW in the absence and presence of inhibitor (RDF) are shown in Figure 2 and electrochemical parameters value are given in Table 2. It is observed from the Table 2 that when mild steel is immersed in SOWW the corrosion potential was –831 mV *vs.* SCE. When 10% of RDF extract was added to the above system the corrosion potential shifted to the anodic side –806 mV *vs.* SCE. This indicates that the anodic reaction is controlled predominantly. However, the shift is within 85 mV. This indicates that the inhibitor behaves as a mixed type of inhibitor controlling both anodic reaction and cathodic reaction to an equal extent by forming a protective film. Further, the LPR value increases from 501 Ohm·cm^2 to 1394 Ohm·cm^2. The corrosion current value decreases from $7.688\cdot10^{-5}$ A/cm^2 to $1.587\cdot10^{-5}$ A/cm^2. Hence, polarization study confirms the formation of a protective film on the metal surface. Thus electrochemical studies are very useful in investigating corrosion protection studies [23–25]. The inhibition efficiency calculated from polarization study comes to 64.06%. The slight difference in inhibition efficiencies between the weight loss method and polarization method may be attributed to the fact, that weight loss method is an average method after immersion period of one day, polarization method is an instantaneous method [26].

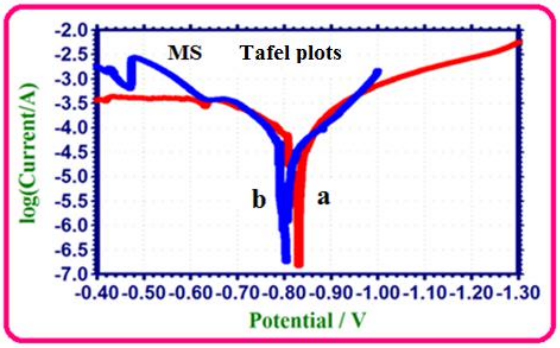

Figure 2. Polarization curves of mild steel immersed in various test solutions (a) SOWW, (b) SOWW + inhibitor (RDF).

Table 2. Corrosion parameters of MS immersed in SOWW in the absence and presence of an aqueous extract of (RDF) obtained by polarization study.

System	**E_{corr}, mV *vs.* SCE**	**b_c, mV/decade**	**b_a, mV/decade**	***LPR*, Ohm·cm^2**	**I_{corr}, A/cm^2**	***IE*%**
SOWW	–831	150	216	501	$7.688\cdot10^{-5}$	–
SOWW + 10% RDF	–806	126	85	1394	$1.587\cdot10^{-5}$	64.06

Analysis of SEM

To establish whether corrosion inhibition is due to the formation of a protective film on the metal surface, SEM images were recorded [27]. Figure 3 (a, b, c) shows the SEM images of polished mild steel, polished mild steel immersed in SOWW (blank) medium and polished mild steel immersed in SOWW containing the inhibitor (RDF) respectively. The image of the polished mild steel surface smooth and image of the blank specimen exhibited rough surface [28, 29] with pit like appearance confirming that the metal has undergone dissolution whereas the smooth surface shown by the inhibitor (10% RDF) system. Thus the SEM study reveals that the mild steel is protected by the formation of non-porous smooth thin film in the presence of inhibitor (RDF).

Figure 3. SEM image of (a) polished mild steel; (b) mild steel immersed in SOWW (blank); (c) mild steel immersed in SOWW containing inhibitor (RDF).

Analysis of AFM

Atomic force microscopy is a powerful technique for gathering of roughness statistics from a variety of surfaces [30]. The two dimensional (2D) and three dimensional (3D) AFM images of polished mild steel, mild steel immersed in SOWW medium and mild steel immersed in SOWW containing the inhibitor (10% RDF) are shown in Figure 4 (a, b, c) and Figure 5 (a, b, c) respectively. AFM images was performed to obtain the average roughness (R_a), root mean square roughness(R_q) and the maximum peak-to-valley (P–V) height values [31]. R_q is much more sensitive than R_a to large and small height deviations from the mean [32]. Table 3 is the summary of the R_q, R_a and (P–V) height values for mild steel surface immersed in different environments.

The value of R_q, R_a and (P–V) height for the polished mild steel surface are 116.89 nm, 98.65 nm and 477.84 nm respectively, which shows a homogeneous surface with some places in which the height is lower than the average depth. Figure 4a and 5a displays the uncorroded polished metal surface. The roughness is due to atmospheric corrosion. The R_q, R_a and (P–V) height values for the mild steel surface immersed in SOWW are 623.84 nm, 456.15 nm and 2901.5 nm respectively. This suggests that mild steel surface immersed in SOWW has a greater surface roughness than the polished mild steel surface. This shows that the unprotected mild steel surface is rougher and is due to the corrosion of the mild steel in SOWW. Figure 4b and 5b displays the corroded metal surface with few pits. The presence of inhibitor (10% RDF) in SOWW reduces to R_q, R_a and (P–V) height values, 199.73 nm, 154.58 nm and 931.49 nm respectively. These parameters confirm that the surface appears smoother. Figure 4c and 5c displays the smooth surface. The smoothness of the surface is due to the formation of a compact protective film of Fe^{2+}–RDF complex on the metal surface thereby inhibiting the corrosion of mild steel [33].

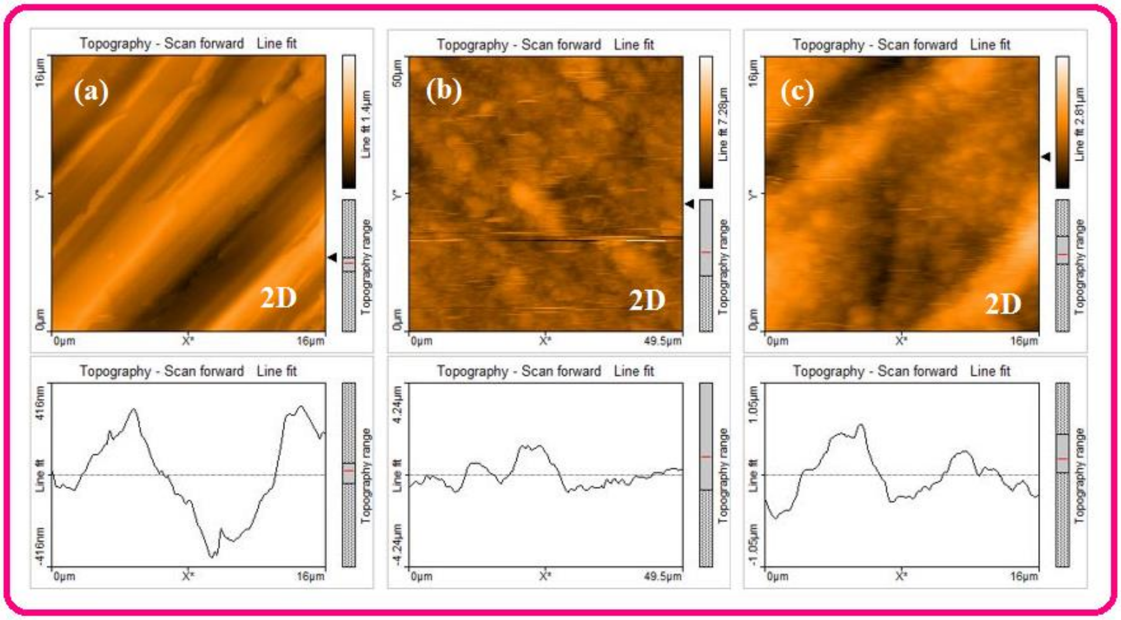

Figure 4. Two dimensional AFM images of the surface (a) polished MS; (b) MS immersed in SOWW; (c) MS immersed in SOWW containing inhibitor (RDF) system.

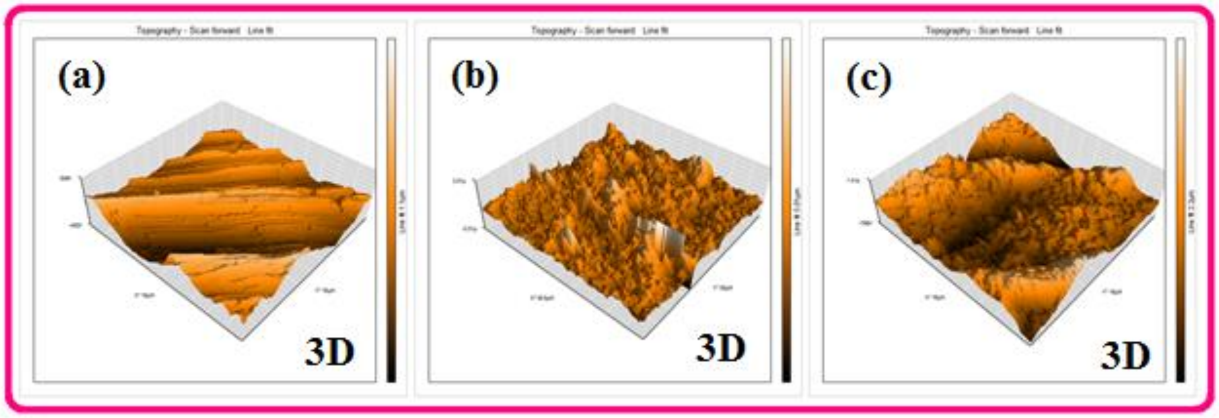

Figure 5. Three dimensional AFM images of the surface (a) polished MS; (b) MS immersed in SOWW; (c) MS immersed in SOWW containing inhibitor (RDF) system.

Table 3. AFM parameters of mild steel surface in the presence and absence of inhibitor (RDF) system.

Sample	RMS (R_q) Roughness (nm)	Average (R_a) Roughness (nm)	Maximum (P–V) height (nm)
Polished MS	116.89	98.65	477.84
MS immersed in SOWW	623.84	456.15	2901.5
MS immersed in SOWW and 10% of RDF extract	199.73	154.58	931.49

Conclusions

The conclusions of the present study are as follows.

- The weight loss study reveals that 96% inhibition efficiency is observed in controlling corrosion of mild steel in SOWW containing an aqueous extract of RDF.
- Potentiodynamic polarization study reveals that the inhibitor system functions as a mixed type inhibitor.
- SEM and AFM study confirmed that a protective film formed on the metal surface.

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