Chemistry and Materials Research
ISSN 2224- 3224 (Print) ISSN 2225- 0956 (Online)
Vol.7 No.4, 2015

Investigation of Salvadora persica Roots Extract as Corrosion Inhibitor for Mild Steel in 1 M HCl and in Cooling Water

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Abstract

Plant extracts have become important as an environmentally acceptable, readily available and renewable source for wide range of inhibitors. Therefore, inhibition of the corrosion of mild steel in hydrochloric acid solution and cooling water was carryout by the root extract of Miswak (Salvadora persica) has been studied using weight loss, electrochemical impedance spectroscopy and potentiodynamic polarization techniques. Inhibition was found to increase with increasing concentration of the extract. The effect of immersion time on the corrosion behavior of mild steel in 1 M HCl and cooling water with addition of extract was also studied. The adsorption of the extract on the mild steel surface obeyed the Langmuir adsorption isotherm. Values of inhibition efficiency calculated from weight loss, potentiodynamic polarization, and electrochemical impedance spectroscopy (EIS) are in good agreement. Polarization curves showed that root extract behaves as a mixed-type inhibitor in both hydrochloric acid and cooling water. The adsorbed film on mild steel surface containing extract was also measured by scanning electron microscopy (SEM) and atomic force microscopy (AFM). The results obtained showed that the root extract could serve as effective inhibitor of the corrosion of mild steel in both hydrochloric acid media and cooling water media.

Keywords: Miswak, Corrosion inhibitor, Cooling water, SEM and AFM.

1. Introduction

The corrosion of metallic materials in acidic solution causes considerable costs. In order to reduce the corrosion of metals, several techniques have been applied.**[A. Sudheer et al, 2011]** A considerable amount of interest has been generated in the study of organic compounds as corrosion inhibitors owing to their usefulness in several industries: during the pickling of metals, cleaning of boilers, acidification of oil wells, etc.**[Sk. A. Ali et al, 2003 and V.S. Sastri 1998]** Now the development of corrosion inhibitors of natural source and nontoxic type has been considered more important and desirable extracts of plant materials contain a wide variety of organic compounds most of them contain hetero atoms such as P, N, S, O. These atoms coordinate with the corroding metal atom (their ions), through their electrons. Hence protective films are formed on the metal surface and hence corrosion is prevented.**[L. Herrag et al, 2010 and Sh. Pournazari et al, 2013]** Salvadora persica (Arak tree) is traditionally used for the treatment of oral infections and the young roots, stems and branches are used as toothbrush.**[R. I. Al Sadhan et al, 1999 and K. Almas et al, 1995]** The Salvadora persica is the most common source of chewing sticks which is used in Africa, South America, Asia and the Middle East. It has different names in different societies, for instance, Miswak, siwak, or arak. Pharmacological studies indicated that Salvadora persica plant has a number of proven medicinal applications and almost all parts (leaves, root bark, fruits and seeds) have been found to be medical activities. **[K. Almas, 1993 and G. Bos, 1993]**

In the present work, we focused on anti-corrosion activity of Miswak extract as a cheap and environment friendly corrosion inhibitor for C-steel in 1 M HCl and cooling water solution by potentiodynamic polarization and electrochemical impedance spectroscopy. Meanwhile, Surface morphology of the metal surface was studied using SEM and AFM. Langmuir adsorption isotherm was drawn and analyzed.

2. Experimental method

The roots of Arak tree (Salvadora persica) were collected from Saudi Arabia. The dried roots (10 g) were cut into small pieces and were soaked in distilled water (500 mL) and refluxed for 5 h. The refluxed solution was filtered to remove any contamination and the aqueous solution was concentrated to 100 mL which left to dry at 50 ^{o}C. The dried extract was used to prepare the solutions of different concentrations (ppm) in order to study the corrosion inhibition properties of Salvadora persica (Miswak).**[V. Sribharathy et al, 2011].**

2.1. Solutions

Solutions of 1M HCl were prepared from commercial analytical reagent using distilled and deionized water. Cooling water solution supplied from of South Fertilizer Company-Basra-Iraq.

2.2. Weight loss measurements

The mild steel sheets used in this present work have the composition presented in Table 1 and strip of 3.5 cm 2.5 cm 0.4 cm size. Before measurements, the mild steel coupons were mechanically polished with series of emery paper of variable grades starting with the coarsest and proceeding in steps to the finest (600) grade, degreased with absolute ethanol, dipped into acetone and washed with deionized water. The coupons were dried and kept in desiccators. After weighing accurately, the specimens were divided into two groups, the first group immersed in 50ml of cooling water and the second immersed in 50ml 1M HCl, without and with addition of different concentrations of Miswak inhibitor at different times (1-5)hrs, the strips were taken out washed, dried and weighed accurately. Duplicate experiments were performed in each and the mean value of the weight loss was reported. Corrosion rate, inhibition efficiency *%IE* and surface coverage (θ) were determined.**[T. Ghailane et al, 2013]**

Table 1: Percentage composition of mild steel alloy

Mn	Ni	P	C	Cu	S	Fe
0.85	**0.07**	**0.05**	**0.3**	**0.21**	**0.041**	**remainder**

The parameters used for the present study are given below:
1. Time: 1, 2, 3, 4 and 5 hrs.
2. Concentration of the inhibitor: 10, 20, 30, 40 and 50 ppm.
3. Temperature: 25 ^{o}C.

2.3. Electrochemical measurements

The assays were performed at room temperature using a three electrode electrochemical cells containing a carbon steel working electrode with a 1 cm^2 surface area, a platinum auxiliary electrode and a saturated calomel electrode (Reference electrode),**[A. S. Abdul Nabi et al, 2013]** Galvanostatic polarization studies were carried out using Bank ELEIKTRONIK INTELLGENT CONTROLS Model MLab 200-Chemistry Department - Education College - Basrah University, for calculation of electrochemical parameters.

2.4. Impedance measurements

Impedance measurements were carried out on mild steel coupons similar to the Tafel experiments. A graphite rod functioned as the auxiliary electrodes. Both of the electrodes were connected to the Electrochemical

Impedance Spectroscopy system (EG and G Electrochemical Impedance Analyzer model 6310 (Germany)), Russia, used to study AC impedance. The experiments were controlled by manufacturer software power suite. The mild steel in the test solution was immersed for 30 min before the frequency scan. Impedance measurements were performed at open circuit potential (E_{ocp}) over a frequency range of 100 kHz–100 MHz with a 10 mV peak-to-peak amplitude using the AC signal. The inhibition efficiencies ($\eta_{EIS}\%$) for each inhibitor concentration were calculated using the following equation**[M. Elayyachy et al, 2006]**:

$$\eta_{EIS}\,(\%) = \frac{R_{ct}-R^{o}_{ct}}{R_{ct}}\times 100$$

where R_{ct} and R^{o}_{ct} are the charge transfer resistances in the presence and absence of the inhibitor, respectively. The values of interfacial double layer capacitance (C_{dl}), were estimated from the impedance value using Nguist plot by the formula**[M. Elayyachy et al, 2006]**:

$$|Z| = \frac{1}{2\pi f C_{dl}}$$

2.5. Atomic force microscopy

The mild steel strips of 1.0 cm x1.0 cm x 0.025 cm sizes were prepared. After immersion in 1 M HCl with and without addition of 30 ppm and in cooling water with and without addition of 50 ppm of Miswak extract at 25 ^{o}C for 48 hrs, the specimen was cleaned with distilled water, dried and then used for AFM.

2.6. Scanning electron microscopy

The specimens used for surface morphological examination were immersed in acid and cooling water containing optimum concentration of inhibitor for 48 hrs. Then they were removed, rinsed with double distilled water and dried. The analysis was performed on Geneziz-model 2000 XMS.

3. Results and discussion

3.1. Weight Loss Measurements

The results of the measurements are shown in Table 2 and 3 for the corrosion of mild steel in cooling water and 1M HCl in the absence and in the presence of different concentrations of Miswak at the temperature of 25°C. The percentage of inhibition efficiency *%IE*, surface parameter coverage θ which represents the part of the surface covered by the inhibitor molecules and corrosion rate were calculated using the following equations**[E.M. Mabrouk et al, 2011]**:

$$\%IE = \left[\frac{W_{corr} - W_{corr(inh)}}{W_{corr}}\right] \times 100$$

$$\theta = \frac{W_{corr} - W_{corr(inh)}}{W_{corr}}$$

where $W_{corr(inh)}$ and W_{corr} are the weight loss values in the presence and in the absence of inhibitor, respectively.

$$R_{corr.} = \frac{\Delta W * K}{A * D * T}$$

Where ΔW= weight losses of metal (gram), K= constant ($5.34\text{x}10^{5}$), A= sample area (cm^2), D= metal density (g/cm^3) and T= exposed time (hr)

Figures 1 and 2 represent the variation of the inhibition efficiency *%IE* as function of the time. The inhibition efficiency increased with increasing the inhibitor concentration.

Table 2: Effect of Miswak on Dissolution Mild steel in 1M HCl

Conc. (ppm)	Blank			10			20			30			40			50		
Time (hr)	Corr. Rate	%IE	Θ	Corr. Rate	%IE	θ	Corr. Rate	%IE	θ	Corr. Rate	%IE	θ	Corr. Rate	%IE	θ	Corr. Rate	%IE	θ
1	92.80			68.33	10.26	0.1026	71.39	23.08	0.2308	59.49	35.90	0.3590	54.49	41.28	0.4128	54.73	41.03	0.4103
2	107.08			64.42	26.67	0.2667	58.30	45.56	0.4556	53.54	50.00	0.5000	51.16	52.22	0.5222	51.16	52.22	0.5222
3	130.88			46.20	56.97	0.5697	51.56	60.61	0.6061	46.00	64.85	0.6485	46.00	64.85	0.6485	49.18	62.42	0.6242
4	165.38			42.46	68.71	0.6871	52.35	68.35	0.6835	45.81	72.30	0.7230	54.14	67.27	0.6727	54.14	67.27	0.6727
5	169.42			49.59	64.33	0.6433	57.59	66.01	0.6601	48.07	71.63	0.7163	57.59	66.01	0.6601	60.92	64.04	0.6404

Table 3: Effect of Miswak on Dissolution Mild steel in cooling water

Conc. (ppm)	Blank			10			20			30			40			50		
Time (hr)	Corr. Rate	%IE	θ	Corr. Rate	%IE	θ	Corr. Rate	%IE	θ	Corr. Rate	%IE	θ	Corr. Rate	%IE	θ	Corr. Rate	%IE	θ
1	66.63			44.90	17.86	0.1786	45.21	32.14	0.3214	40.45	39.29	0.3929	42.83	35.71	0.3571	30.93	53.57	0.5357
2	73.77			41.97	30.65	0.3065	41.64	43.55	0.4355	38.07	48.39	0.4839	40.45	45.16	0.4516	32.12	56.45	0.5645
3	77.73			40.35	36.73	0.3673	36.49	53.06	0.5306	34.11	56.12	0.5612	32.52	58.16	0.5816	30.14	61.22	0.6122
4	79.72			35.63	45.52	0.4552	33.31	58.21	0.5821	30.34	61.94	0.6194	29.15	63.43	0.6343	19.63	75.37	0.7537
5	81.38			30.85	53.80	0.5380	31.89	60.82	0.6082	28.08	65.50	0.6550	25.22	69.01	0.6901	13.80	83.04	0.8304

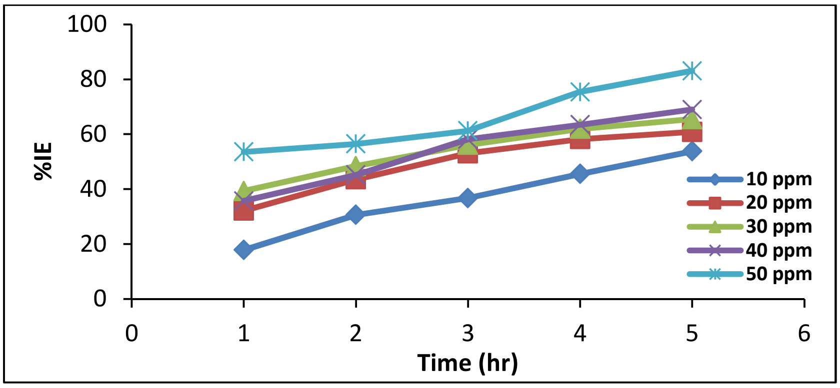

Figure 1: Variation of the Inhibition Efficiency %IE as a Function of the Time in the Presence of Different Concentrations of Miswak at 25°C in cooling water

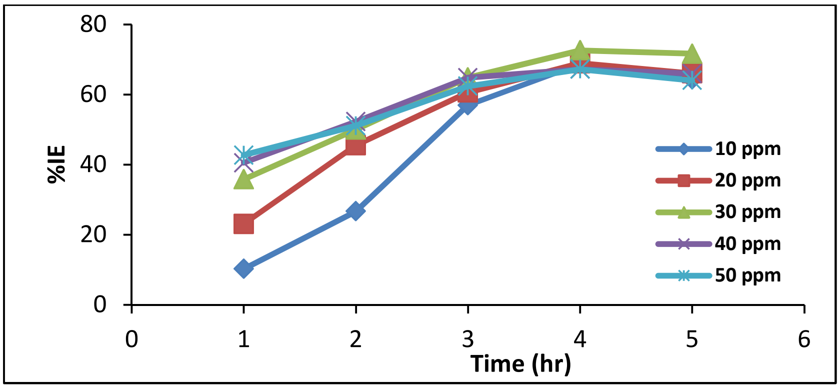

Figure 2: Variation of the Inhibition Efficiency %IE as a Function of the Time in the Presence of Different Concentrations of Miswak at 25°C in 1M HCl

3.2. Adsorption Isotherms

The surface coverage values (θ) calculated from weight loss measurements were fitted into the Langmuir adsorption isotherm**[A. Singh et al, 2010]**. The Langmuir adsorption isotherm model has the form:

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

Where C is the concentration of inhibitor, K_{ads} is the equilibrium constant of adsorption process. A plot of C/θ vs C is linear, as shown in Figures 3 and 4. The slope deviates from unity indicating that there is attraction or repulsion in the adsorbed layer of inhibitor on the steel surface.**[A. I. Onen et al, 2011]** Equilibrium constant of adsorption K_{ads} in HCl solution is 193 and R^2=0.995, while e K_{ads} in cooling water is 33.65 and R^2=0.961.

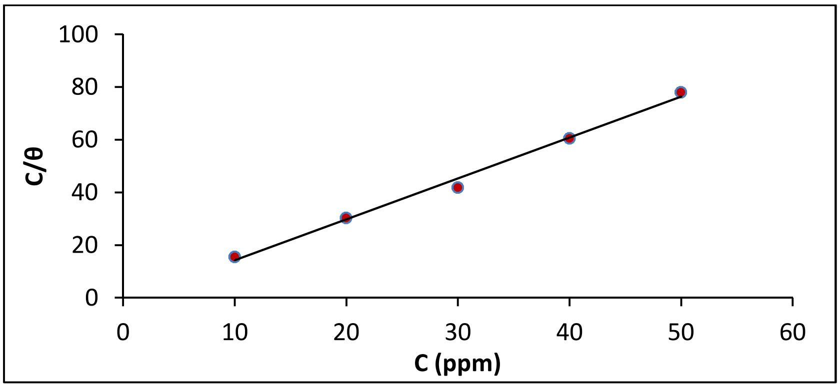

Figure 3: Langmuir adsorption isotherm of Miswak inhibitor in 1M HCl at 25 °C

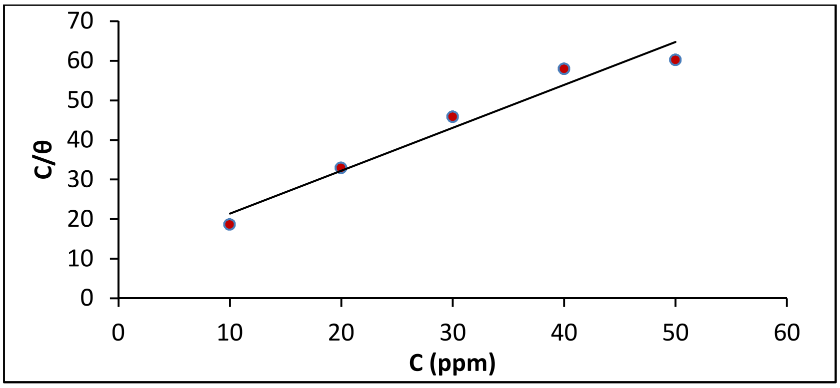

Figure 4: Langmuir adsorption isotherm of Miswak inhibitor in cooling water at 25 °C

3.3. Polarization Measurements

Polarization behavior of mild steel in cooling water and 1M HCl in the presence and absence of Miswak is shown in Figures 5 and 6, respectively. Tables 4 and 5 gives values of corrosion current density (I_{corr}), corrosion potential (E_{corr}), cathodic Tafel slope (β_c), anodic Tafel slope (β_a) . The inhibition efficiency ($IE\%$) is calculated by relation(17):

$$E\% = \left[\frac{I_{corr} - I_{corr(inh)}}{I_{corr}}\right] \times 100$$

where I_{corr} and $I_{corr(inh)}$ are the corrosion current in the absence and in the presence of inhibitor, respectively.

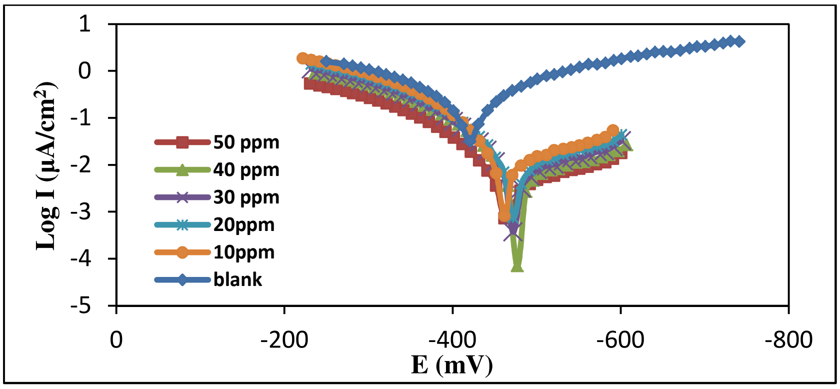

Figure 5: Tafel curves of Mild steel in the cooling water in the absence and presence of different concentrations of Miswak inhibitor at 25°C

Table 4: The values of corrosion parameters for the corrosion of mild steel in cooling water by galvanostatic polarization in presence Miswak

Inhibitor Concentration (ppm)	i_{corr} $\mu A/cm^2$	E_{corr} mVolt	βc mV/dc	βa mV/dc	%IE
Blank	198	-410.4	113.1	95.2	
10	75.7	-432.5	123.5	67.5	61.76
20	66.7	-438.9	125.9	63.2	66.31
30	60.4	-472.2	119.7	58.1	69.49
40	50.3	-461.3	128.2	65.7	74.59
50	40.5	-468.9	127.1	61.6	79.54

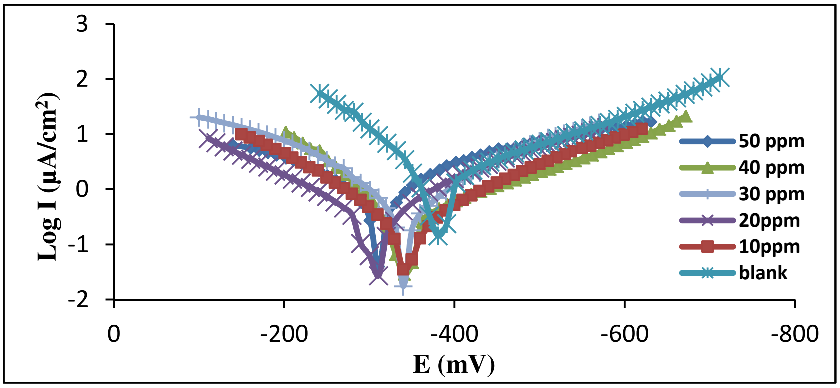

Figure 6: Tafel curves of mild steel in 1M HCl solution in the absence and presence of different concentrations of Miswak inhibitor at 25°C

Table 5: The values of corrosion parameters for the corrosion of mild steel in 1M HCl by galvanostatic polarization in presence Miswak

Inhibitor Concentration (ppm)	icorr µA/cm2	Ecorr mVolt	βc mV/dc	βa mV/dc	%IE
Blank	225.3	-382.6	-113.1	95.2	
10	101.2	-343.9	-94.5	97.6	55.08
20	85.7	-340.8	-76.2	99	61.96
30	61.5	-350.8	-91.1	94.6	72.70
40	74.2	-355.6	-103.9	89.6	67.06
50	76.2	-357.2	-97.4	94.7	66.17

As it is shown in Figure 5 and Table 4, cathodic polarization curves of cooling water rise to parallel Tafel lines indicating that the hydrogen evolution reaction is control activation. Thus the presence of Miswak does not affect the mechanism of this process. The addition of Miswak causes a decrease of current density. Cathodic Tafel slope (β_c) do not change when the concentration increase .The result demonstrate that the hydrogen reduction is inhibited and that the inhibition efficiency increases with inhibitor concentration to reach a maximum value of 72.7% at optimum concentration of 50 ppm of Miswak . From Figure 6 and Table 5, the polarization curves of mild steel in 1M HCl with and without Miswak show that the presence of inhibitor decreases the current density and inhibition efficiency reaches to a maximum value 72.70% at optimum concentration of 30 ppm.
This fact means that Miswak inhibits anodic reaction and acts as a mixed type inhibitor.

3.4. Electrochemical impedance spectroscopy

Figures 7 and 8 illustrate the Nyquist diagrams for mild steel immersed in tested solution (cooling water and 1 M HCl, respectively) in the absence and presence of different concentrations of Miswak at 25°C. The impedance spectra exhibit one single capacitive loop, which indicates that the corrosion of steel is mainly controlled by a charge transfer process.**[M. A. Quraishi et al, 2009]** The AC impedance parameters such as charge transfer resistance (R_t) and double layer capacitance (C_{dl}) are given in Tables 6 and 7. When mild steel is immersed in cooling water, the charge transfer resistance R_t is 300 Ω; the double layer capacitance C_{dl} is 530.79 µF/cm^2, while in acid, the charge transfer resistance R_t is 77 Ω; the double layer capacitance C_{dl} is 2068 µF/cm^2. When the inhibiter is added, the R_t value increases and C_{dl} value decreases. This confirms that a protective film is formed on the metal surface. This decreases the corrosion rate of mild steel and increases the inhibition efficiency.**[T. Ghailane et al, 2013]**

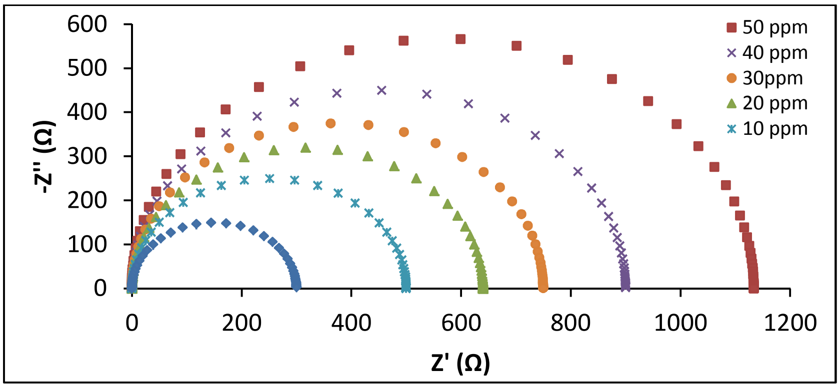

Figure 7: Nyquist plots of mild steel in cooling water with various concentration of Miswak at 25°C

Table 6: Electrochemical impedance parameters for mild steel in cooling water in the various concentrations Miswak at 25°C

Inhibitor Concentration (ppm)	R_p	C_{dl}	%E
Blank	300	530.79	
10	500	318.47	40.00
20	640	248.81	53.13
30	750	212.31	60.00
40	900	176.93	66.67
50	1134	140.42	73.54

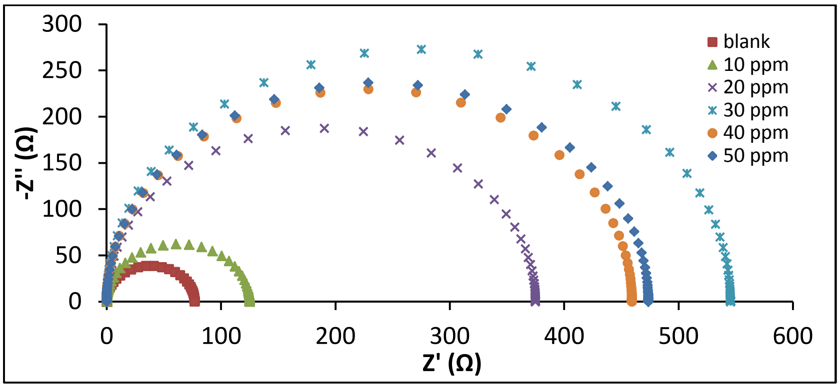

Figure 8: Nyquist plots of mild steel in 1M HCl with various concentration of Miswak at 25°C

Table 7: Electrochemical impedance parameters for mild steel in 1M HCl in the various concentrations Miswak at 25°C

Inhibitor Concentration (ppm)	Rp	Cdl	%E
Blank	77	2068.00	
10	125	1273.88	38.40
20	375	424.63	79.47
30	545	292.18	85.87
40	459	346.91	83.22
50	473	336.65	73.57

3.5. SEM analyses

Scanning electron microscopy analysis was used to determine the morphology of the mild steel specimens before and after immersed in tested solution (cooling water and 1 M HCl) for 48 hrs at 25 °C in the absence and presence of optimum concentration of Miswak inhibitor compared with the freshly polished surface). The specimen surface in the absence of the inhibitors (Figure 9b & 9c) is strongly corroded, and the surface becomes porous and rough. In the presence of inhibitor (Figure 10a and 10b), the surface of the specimens are well protected and the photographs are almost the same as that of the freshly polished surface (Figure 9a). These results indicate that a good protective adsorption film could be formed on the specimen surface, and the corrosion of mild steel in cooling water and 1M HCl solution is inhibited remarkably by the inhibitor.**[M. Tourabi et al, 2013]**

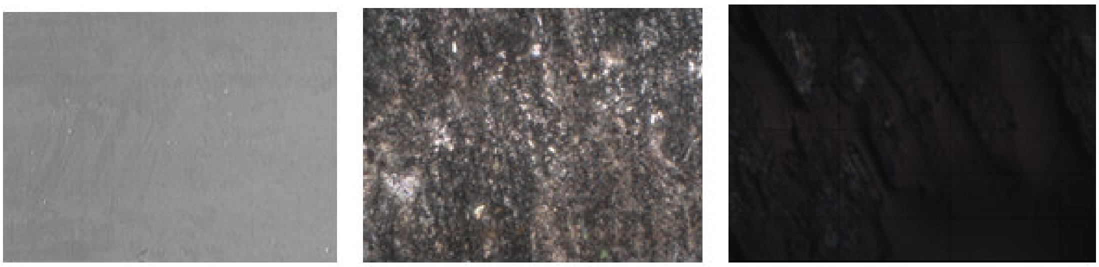
(a) (b) (c)

Figure 9: SEM images of the mild steel surface of (a) polished mild steel (control), (b) mild steel immersed in 1M HCl (blank) and (c) mild steel immersed in cooling water (blank)

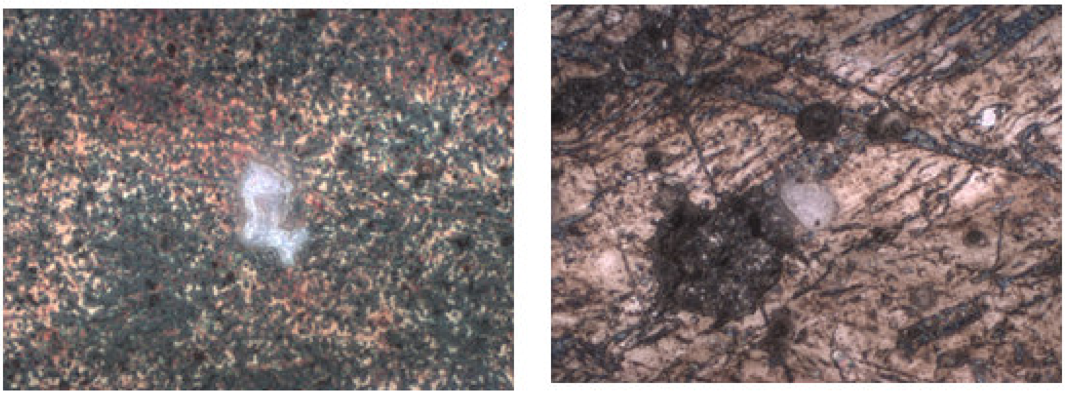
(a) (b)

Figure 10: SEM of mild steel surface after immersion for a period of 48 hrs in (a) 1M HCl and (b) cooling water, in the presence of Miswak inhibitor

3.6. Analysis of AFM images

Figure 11a, 11b and 11c show the AFM images of polished metal, corroded surface in the absence of inhibitor in 1M HCl and in cooling water, respectively. Figures 11d and 11e show the AFM images of smoothened surface in the presence of optimum concentration Miswak inhibitor in 1M HCl and in cooling water, respectively. Table 8 gives the corresponding average roughness R_a and RMS roughness (R_q) values. A comparative view of the above roughness table clearly demonstrates that the surface of the metal is smoothened due to the adsorption layer formed.**[S. Rajendran et al, 2012]**

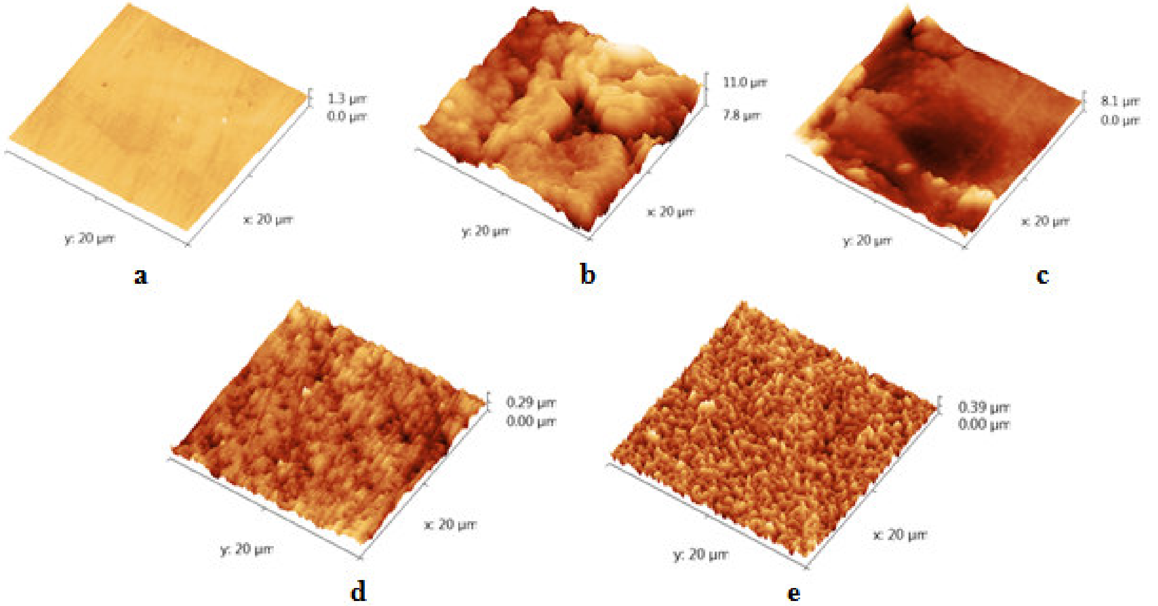

Figure 11: Three dimensional AFM image of mild steel surface, a=fresh, b= immersed in 1 M HCl solution, c= immersed in cooling water, d= immersed in 1 M HCl solution + Miswak and e= immersed in cooling water + Miswak

Table 8: AFM roughness data

Sample	Average (Ra) Roughness (nm)	RMS (Rq) Roughness (nm)
Fresh mild steel (Control)	24	31
Mild steel immersed in cooling water	200	230
Mild steel immersed in cooling water + Miswak	118	128
Mild steel immersed in 1M HCl	400	510
Mild steel immersed in 1M HCl + Miswak	127	135

4. Conclusion

The aqueous extract of Miswak can be used as an excellent corrosion inhibitor for mild steel in acidic medium and cooling water at 25°C. To obtain the maximum protection efficiency, critical plant extract concentration should be determined. The inhibition mechanism depends on the formation of a stable plant extract-complex on the steel surface. Polarization studies reveal that the extracts behave as mixed type inhibitors. A systematic study on plant extracts is necessary to get a complete image of the possibilities to obtain corrosion inhibitors and to determine the most suitable formulation approach. Moreover, the inhibitor obtained from plant extracts could be applied in many fields such as oil pipelines, paints, fuels, metal processing solutions, ..etc.

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