

***Ocimum sanctum* as Inhibitor for Mild Steel Corrosion in Phosphoric Acid Medium**

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The inhibition effect of *Ocimum sanctum* on corrosion of mild steel in phosphoric acid solutions has been studied. The mass loss and polarization techniques were employed at 302-333 K. The inhibition efficiency increased with increase in concentration of inhibitors and decreased with rise in temperature from 302 to 333 K and in acid concentration from 1 to 5 N. The adsorption of this compound on the mild steel surface from the acids has been found to obey Temkin's adsorption isotherm. The potentiostatic polarization results revealed that *Ocimum sanctum* is a mixed type inhibitor. The values of activation energy (E_a), free energy of adsorption (ΔG_{ads}), enthalpy of adsorption (ΔH) and entropy of adsorption (ΔS) were also calculated. The plots of W_f against time (d) at 302 K gives straight line, which suggested that it obeys first order kinetics and also calculate the rate constant (k) and half life period ($t_{1/2}$).

Key Words: Mild steel, Phosphoric acid, Corrosion inhibition, Temkin's adsorption isotherm, Potentiostatic polarization.

INTRODUCTION

Phosphoric acid is a major chemical product which has many important uses especially in the production of fertilizers. Most of the acid is produced from phosphate rock by wet process. Generally nickel-base alloys and stainless steel are frequently used in many parts of the wet process and a considerable quantity of data has been published about the resistance of these materials to corrosion by phosphoric acid solution¹⁻⁵. Most of the previous studies were focused on the inhibition of stainless steel or chromium-nickel steel in HCl or H₂SO₄ solutions using organic compounds containing nitrogen, sulfur and oxygen atoms as corrosion inhibitors^{6,7}. Mild steel is extensively used in industries especially for structural applications. But its susceptibility to rusting in humid air and its high dissolution rate in acidic medium are the major obstacles in its use on a large scale. Hence, the study of corrosion

inhibition of mild steel in aqueous aggressive media is very important. Different nitrogen containing organic compounds have been studied as corrosion inhibitors for mild steel⁸⁻¹⁰. Organic inhibitors are widely used in various industries. Among them, heterocyclic compounds comprise a potential class of inhibitors. There is wide consideration in the literature regarding corrosion inhibition studies by nitrogen containing heterocyclics¹¹⁻¹⁸. Compounds containing nitrogen and sulphur atoms are of particular importance as they often provide excellent inhibition compared with compounds containing only nitrogen or sulphur¹⁹⁻²⁴. The corrosion inhibiting property of these compounds is attributed to their molecular structure. These compounds contain π electrons and heteroatoms, which induce greater adsorption of the inhibitor molecules onto the mild steel surface.

The aim of the present investigation was to examine the inhibitive action of *Ocimum sanctum* extract towards the corrosion of mild steel in 1 and 5 N phosphoric acid by mass loss and potentiostatic polarization methods at 302-333 K.

EXPERIMENTAL

Mass loss measurement: Before extraction, leaves of the plant (*Ocimum sanctum*) were prepared by drying at room temperature (27 °C) followed by grinding. The dried material was extracted successively with methanol in Soxhlet extractor for 24 h at 60 °C. The extracted plant material was filtered and methanol filtrate was evaporated to 40 °C. The residue was dissolved in hot water and it was used as inhibitor.

Mild steel specimens were cut to the size of 5 cm × 1 cm from the mild steel sheets having the following percentage composition Fe = 99.686, Ni = 0.013, Mo = 0.015, Cr = 0.043, S = 0.014, P = 0.009, Si = 0.007, Mn = 0.196, C = 0.017. Mass loss measurements were performed as per ASTM method described previously^{19,25,26}. Mass loss measurements were carried out in 1 and 5 N concentration of phosphoric acid with inhibitor (*Ocimum sanctum*) in the concentration range of 0.02 to 0.1 % at 302 to 333 K for an immersion period of 5 h with and without inhibitor. At 302 K, the immersion period of 24, 48, 72 and 96 h were also studied. All the solutions were prepared using AR grade chemicals with double distilled water.

Potentiostatic polarization measurement: Polarization measurements were carried out in a conventional three-electrode cell assembly. Mild steel strips of same composition coated with lacquer with an exposed area of 1 cm² were used as working electrode. The saturated calomel electrode and the platinum foil were used as reference and counter electrode, respectively. The potentiostatic polarization was carried out using BAS-100A model instrument and the experiments were carried out at 302 to 333 K.

RESULTS AND DISCUSSION

Mass loss studies: Table-1 shows the values of inhibition efficiency (IE %), surface coverage (θ) and corrosion rate obtained for different concentrations of these inhibitor in 1 and 5 N phosphoric acid for immersion period of 5 h at 302-333 K. From the mass loss value, the inhibition efficiency (IE %) and surface coverage (θ) were calculated using the following equation^{27,28}:

$$IE \% = \frac{W_u - W_i}{W_u} \times 100 \quad (1)$$

$$\theta = \frac{W_u - W_i}{W_u} \quad (2)$$

where W_u and W_i are the corrosion rates for mild steel in the absence and presence of inhibitor respectively at the same temperature.

TABLE-1
CALCULATED CORROSION RATE, INHIBITION EFFICIENCY (IE %) AND SURFACE COVERAGE (θ) VALUES FOR *Ocimum sanctum* FROM MASS LOSS DATA IN 1 AND 5N H₃PO₄

Temp. (K)	Conc. of <i>Ocimum sanctum</i> (%)	1 N			5 N		
		Corrosion rate (mmpy)	Surface coverage (θ)	Inhibition efficacy (IE %)	Corrosion rate (mmpy)	Surface coverage (θ)	Inhibition efficacy (IE %)
302	Blank	6.80	–	–	26.22	–	–
	0.02	1.55	0.7723	77.23	8.75	0.6662	66.62
	0.04	1.20	0.8246	82.46	6.71	0.7440	74.40
	0.06	0.67	0.9025	90.25	3.81	0.8547	85.47
	0.08	0.21	0.9690	96.90	1.90	0.9277	92.77
	0.10	0.10	0.9857	98.57	1.41	0.9464	94.64
313	Blank	11.93	–	–	70.22	–	–
	0.02	3.08	0.7420	74.20	26.25	0.6261	62.61
	0.04	2.39	0.7998	79.98	20.04	0.7146	71.46
	0.06	1.42	0.8814	88.14	13.71	0.8047	80.47
	0.08	0.70	0.9412	94.12	8.22	0.8829	88.29
	0.10	0.49	0.9589	95.89	6.60	0.9061	90.61
333	Blank	19.82	–	–	157.39	–	–
	0.02	6.99	0.6476	64.76	78.24	0.5029	50.29
	0.04	5.55	0.7199	71.99	58.30	0.6296	62.96
	0.06	3.95	0.8010	80.10	42.26	0.7315	73.15
	0.08	2.45	0.8766	87.66	28.11	0.8214	82.14
	0.10	2.05	0.8967	89.67	20.51	0.8697	86.97

It clearly indicates that addition of inhibitor to the acid has reduced the corrosion rate. The inhibition efficiency increased with increase in concentration of inhibitor and decreased with rise in temperature from 302 to 333

K and in acid concentration from 1 to 5 N. The values of corrosion rate and inhibition efficiencies of the inhibitor were found to depend on the molecular structure of inhibitor. The maximum inhibition efficiency of *Ocimum sanctum* (conc. 0.1 %) was found to be 98.57 % in 1 N phosphoric acid and 94.64 % in 5 N phosphoric acid (Table-1).

Thermodynamics/Kinetics consideration: Table-2 shows the calculated values of activation energy E_a (KJ/mol), free energy of adsorption ΔG_{ads} (KJ/mol), enthalpy of adsorption ΔH (KJ/mol), entropy of adsorption ΔS (KJ mol⁻¹ K⁻¹), rate constant k (s⁻¹) and half-life period $t_{1/2}$ (s) for mild steel corrosion in 1 and 5 N phosphoric acid with and without inhibitor. Energy of activation (E_a) has been calculated from the slopes of plots of $\log p$ vs. $1/T$ in Figs. 1 and 2 for *Ocimum sanctum* and also with the help of Arrhenius equation²⁹⁻³¹.

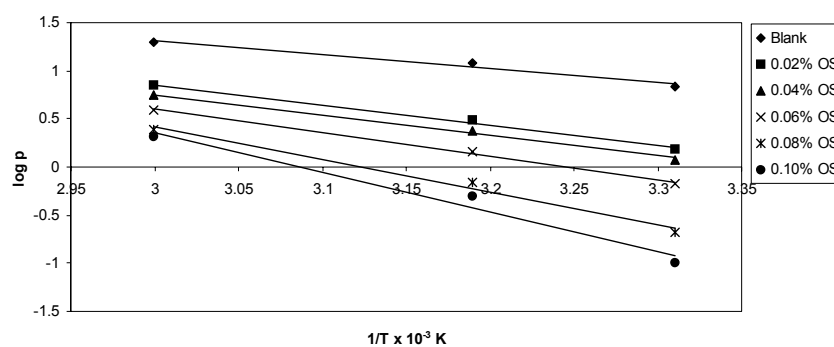


Fig. 1. Arrhenius plot for 1 N H₃PO₄ with *Ocimum sanctum* (OS)

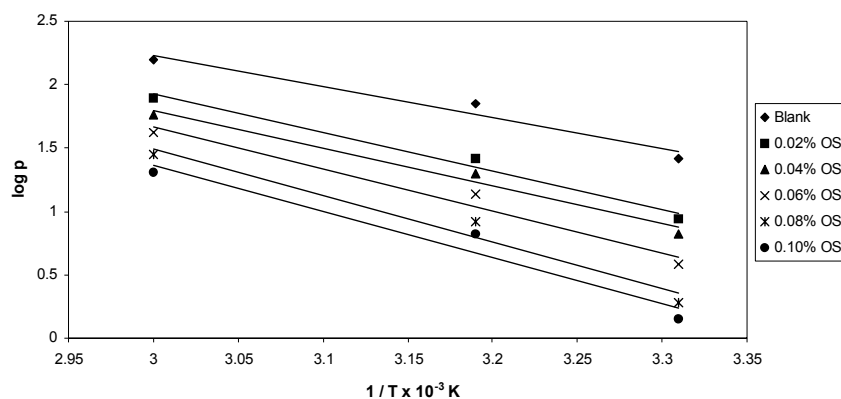


Fig. 2. Arrhenius plot for 5 N H₃PO₄ with *Ocimum sanctum* (OS)

TABLE-2
CALCULATED VALUES OF ACTIVATION ENERGY E_a (KJ/mol), FREE ENERGY OF ADSORPTION ΔG_{ads} (KJ/mol), ENTHALPY OF ADSORPTION ΔH (KJ/mol), ENTROPY OF ADSORPTION ΔS (KJ mol⁻¹ K⁻¹), RATE CONSTANT k (time⁻¹) AND HALF-LIFE PERIOD $t_{1/2}$ (time) FOR MILD STEEL CORROSION IN 1 and 5 N PHOSPHORIC ACID WITH *Ocimum sanctum* (OS)

Inhibitor	Conc. of inhibitor (%)	E_a from eqn. 1 (KJ/mol)	E_a from Plot (KJ/mol)	ΔG_{ads} (KJ/mol)	ΔH (KJ/mol)	ΔS (KJ mol ⁻¹ K ⁻¹)	Rate constant $k \times 10^{-6}$ (s ⁻¹)	Half-life period $t_{1/2}$ (s)	
OS	Blank	28.86	28.72	–	23.87	–	5.73	120934	
	0.02	40.63	41.00	-27.71	35.64	0.1902	4.58	151165	
	+	0.04	41.31	42.10	-28.12	36.32	0.1915	4.19	165586
	1 N	0.06	47.52	48.12	-28.64	42.52	0.2137	3.81	181829
	H ₃ PO ₄	0.08	66.27	65.98	-29.62	61.28	0.2730	3.47	199981
	0.10	81.47	82.02	-30.20	76.47	0.3203	3.25	213080	
OS	Blank	48.34	48.74	–	43.35	–	22.82	30370	
	0.02	58.32	58.90	-26.52	53.33	0.2398	19.72	35139	
	+	0.04	59.10	59.50	-26.91	54.11	0.2433	19.27	35960
	5 N	0.06	64.91	65.12	-27.20	59.92	0.2442	19.00	36461
	H ₃ PO ₄	0.08	72.22	71.99	-27.82	67.23	0.2854	18.69	37092
	0.10	72.68	72.81	-28.05	67.69	0.2875	18.23	38007	

$$\log \frac{p_2}{p_1} = \frac{E_a}{2.303 \times R} \left(\frac{1}{T_1} - \frac{1}{T_2} \right) \quad (3)$$

where p_1 and p_2 are the corrosion rate at temperatures T_1 and T_2 , respectively. E_a values given in Table-2 show that the E_a values for the corrosion of mild steel in 1 and 5 N phosphoric acid are 28.86 and 48.34 KJ/mol, respectively. In acid containing inhibitor, the E_a values are found to be higher than that of the uninhibited system. The higher values of E_a indicate physical adsorption of the inhibitors on metal surface³². The values of E_a calculated from the slopes of Arrhenius plot and by using eqn. 1 are approximately almost similar.

The free energy of adsorption (ΔG_{ads}) at different temperatures was calculated from the following equation³³:

$$\Delta G_{ads} = -RT \ln (55.5 K) \quad (4)$$

and K is given by

$$K = \frac{\theta}{C(1-\theta)} \quad (5)$$

where θ is surface coverage on the metal surface, C is concentration of inhibitor in mol/L and K is equilibrium constant.

From Table-2, the negative values of ΔG_{ads} obtained indicate the spontaneous adsorption of the inhibitor and are usually characteristics of strong interaction with the metal surface. It is found that the ΔG_{ads} values are less

than (less - ve values) - 40 KJ/mol indicating that inhibitors are physically adsorbed on the metal surface^{34,35}.

Free energy of adsorption ΔG_{ads} of *Ocimum sanctum* can be calculated from eqn. 4 at 302 to 333 K while the enthalpy of adsorption ΔH and entropy of adsorption ΔS can be calculated from the following equation.

$$\Delta H = E_a - RT \quad (6)$$

$$\Delta G = \Delta H - T\Delta S \quad (7)$$

ΔS can be easily calculated at 302 to 333 K for the concentration of *Ocimum sanctum*. Table-2 shows thermodynamic data obtained from the inhibitor used in this study. It is clear that the activation energy increases regularly with increasing the efficiency of the inhibitor.

Ideally, a corrosion inhibitor is a substance that greatly increases the activation energy of corrosion. The negative values of ΔG_{ads} indicate the spontaneous adsorption of inhibitor on the surface of mild steel. It is also observed that ΔS is increased with increasing the efficiency of the inhibitors. This is opposite to that we expect, since the adsorption is an exothermic process and is always accompanied by a decrease of entropy. Ateya *et al.*³⁶ have described this situation as the adsorption of the organic compound, which is accompanied by desorption of water molecules off the surface. Thus, while the adsorption process is believed to be exothermic and associated with a decrease in entropy of the solute, the opposite is true for the solvent. Therefore, this gain in entropy that accompanies the substitutional adsorption process is attributed to the increase in solvent entropy.

Figs. 3 and 4 show the relation between the inhibition efficiency and temperature for 0.02 to 0.10 % of *Ocimum sanctum* on mild steel in 1 and 5 N phosphoric acid, respectively. The order of the efficiency is decreased with increasing temperature. These results indicate that the protective film start to break down with increasing temperature.

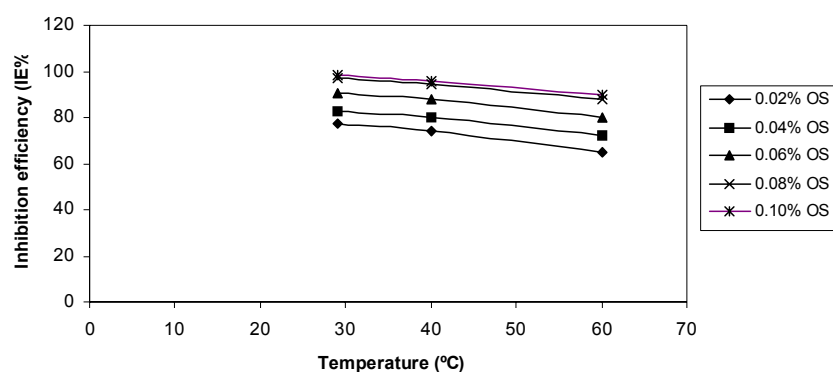


Fig. 3. Relation between the inhibition efficiency and temperature for 0.02 to 0.10 % of *Ocimum sanctum* (OS) on mild steel in 1 N H₃PO₄

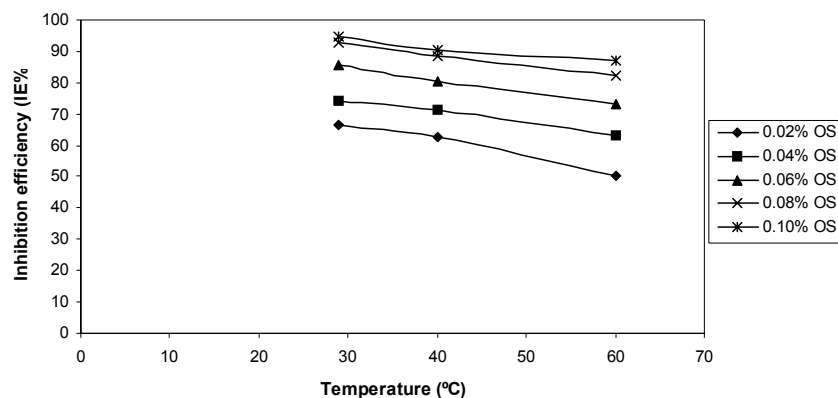


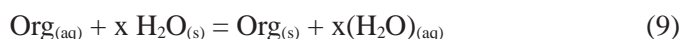
Fig. 4. Relation between the inhibition efficiency and temperature for 0.02 to 0.10 % of *Ocimum sanctum* (OS) on mild steel in 5 N H₃PO₄

The values of rate constant k were evaluated from the plots of $\log W_f$ vs. time (d) in Figs. 5 and 6 for *Ocimum sanctum*. Linear plots were obtained which revealed first order kinetics. The values of half-life period $t_{1/2}$ were calculated using the equation below³⁰:

$$t_{1/2} = \frac{0.693}{k} \quad (8)$$

The rate constant k decreases with increase in concentration of inhibitor where as the half-life increases with concentration of inhibitor²⁷. The adsorption of the organic molecules can affect in several ways, the behaviour of the electrochemical reactions involved in the corrosion process. The action of organic inhibitors also depends on the type of interaction between the substance and the metallic surface. This interaction causes a change either in the electrochemical process mechanism or in the surface available to the process³⁷⁻³⁹.

Adsorption isotherms: The electrochemical process on the metal surface is likely to be closely related to the adsorption of the inhibitor⁴⁰ and the adsorption is known to depend on the chemical structure of the inhibitor^{10,41,42}. The adsorption of the inhibitor molecules from aqueous solution can be regarded as quasisubstitution process¹⁰ between the organic compound in the aqueous phase, $\text{Org}_{(\text{aq})}$ and water molecules at the electrode surface, $\text{H}_2\text{O}_{(\text{s})}$.



where x the size ratio, is the number of water molecules displaced by one molecule of organic inhibitor.

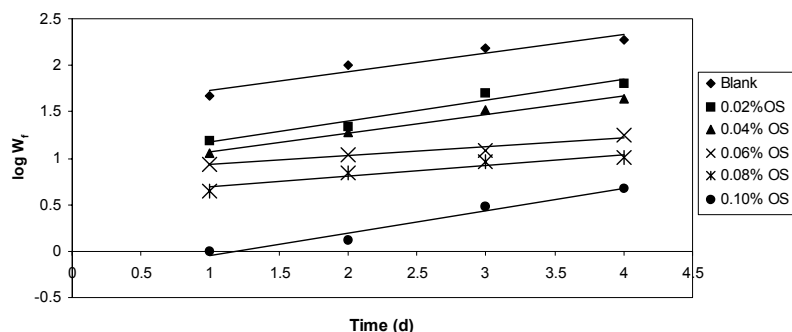


Fig. 5. Plot of $\log W_f$ vs. time (d) for 1 N H_3PO_4 with *Ocimum sanctum* (OS)

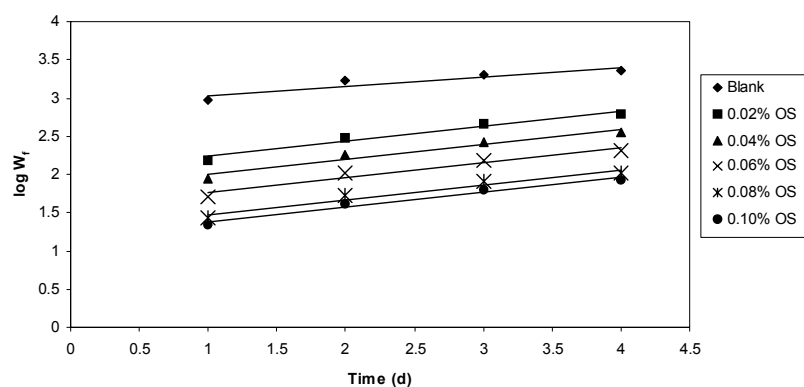


Fig. 6. Plot of $\log W_f$ vs. time (d) for 5 N H_3PO_4 with *Ocimum sanctum* (OS)

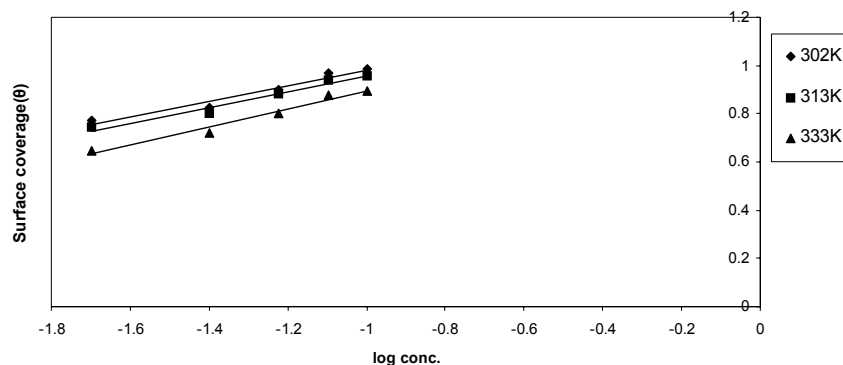
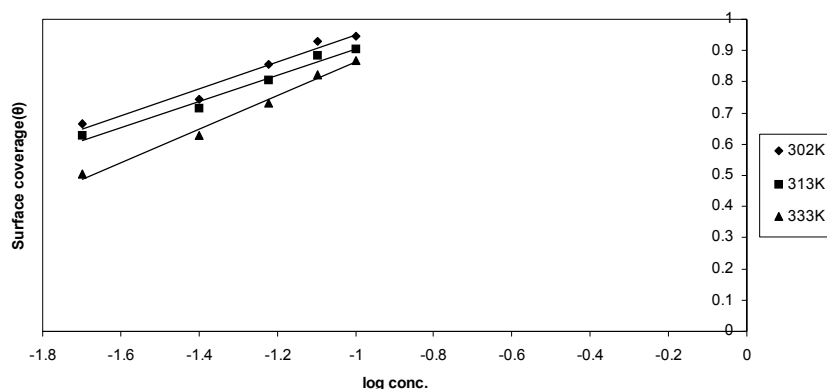
Adsorption isotherms are very important in determining the mechanism of organo-electrochemical reactions. The most frequently used isotherms are those of Langmuir, Frumkin, Parsons, Temkin, Flory-Huggins and Bockris-Sinkles⁴³⁻⁴⁶. All these isotherms are of the general form:

$$f(\theta, x) \exp(-a\theta) = KC \quad (10)$$

where $f(\theta, x)$ is the configurational factor that depends essentially on the physical model and assumptions underlying the derivation of the isotherm⁴⁷.

The mechanism of inhibition of corrosion is generally believed to be due to the formation and maintenance of a protective film on the metal surface.

The plot of surface coverage (θ) obtained by mass loss method vs. $\log C$ for different concentrations of the compound show a straight line indicating that the adsorption of the compounds from acids on mild steel surface follows Temkin's adsorption isotherm³³. This also pointed to corrosion inhibition by these compounds being a result of their adsorption on the metal surface. Figs. 7 and 8 show the Temkin's adsorption isotherm for *Ocimum sanctum*.

Fig. 7. Temkin's adsorption isotherm plot for 1 N H₃PO₄ with *Ocimum sanctum* (OS)Fig. 8. Temkin's adsorption isotherm plot for 5 N H₃PO₄ with *Ocimum sanctum* (OS)

Potentiostatic polarization studies: The polarization behaviour of mild steel functioning as cathode as well as anode in the test solutions is shown in Fig. 9 for 1 N H₃PO₄ at 302 K for *Ocimum sanctum*. Similar curves were also obtained for *Ocimum sanctum* in 1 and 5 N phosphoric acid at 302 to 333 K and the electrochemical data obtained from the studies are shown in Table-3. It is evident that amide bring about considerable polarization of the cathode as well as anode. It was, therefore, inferred that the inhibitive action is of mixed type. The cathodic and anodic Tafel slopes increased with increasing inhibitor concentrations and the increase was predominant in the case of the former indicating that the cathodic inhibition was dominating through the inhibitive active is of mixed nature. The non-constancy of Tafel slopes for different inhibitor concentration reveals that the inhibitor act through their interference in the mechanism of the corrosion processes at the cathode as well as the anode.

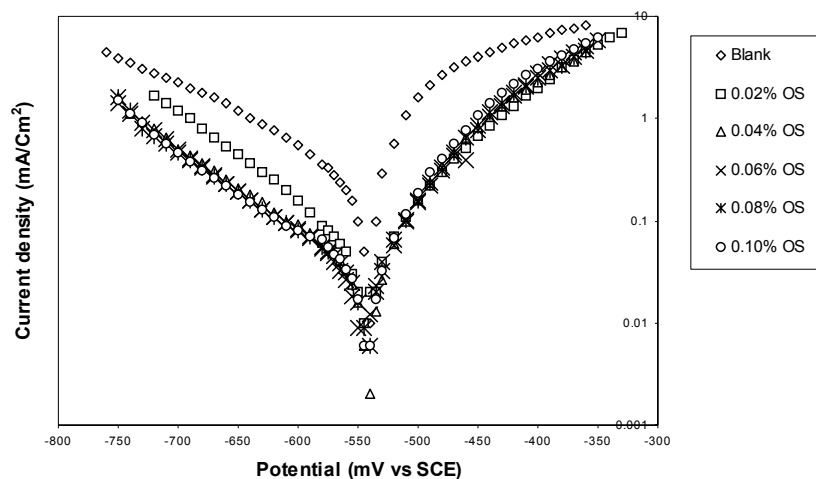


Fig. 9. Typical potentiostatic curves for mild steel in 1 N H_3PO_4 with *Ocimum sanctum* (OS) at 302 K

TABLE-3
ELECTROCHEMICAL POLARIZATION PARAMETERS FOR THE CORROSION
BEHAVIOUR OF MILD STEEL IN 1 AND 5 N PHOSPHORIC ACID IN
ABSENCE AND PRESENCE OF *Ocimum sanctum* (OS) AT 302-333 K

Conc. of inhibitor (%)	1 N					5 N				
	E_{corr} vs. SCE (mV)	i_{corr} $\mu\text{A}/\text{cm}^2$	Tafel constant (mV/decade)		IE (%)	E_{corr} vs. SCE (mV)	i_{corr} $\mu\text{A}/\text{cm}^2$	Tafel constant (mV/decade)		IE (%)
			ba	-bc			ba	-bc		
302 K										
Blank	-543	210	50	130	—	-510	700	50	150	—
0.02 OS	-547	49	45	127	76.67	-556	244	48	140	65.14
0.04 OS	-544	39	42	120	81.43	-547	193	47	138	72.43
0.06 OS	-545	18	40	115	91.43	-542	95	44	134	86.43
0.08 OS	-542	8	40	110	96.19	-535	67	42	130	90.43
0.10 OS	-541	5	39	101	97.62	-530	45	40	127	93.57
313 K										
Blank	-535	680	100	150	—	-505	1000	30	175	—
0.02 OS	-544	177	96	142	73.97	-535	375	29	169	62.50
0.04 OS	-543	146	92	140	78.53	-530	290	27	164	71.00
0.06 OS	-540	71	90	137	89.56	-526	175	28	161	82.50
0.08 OS	-539	46	87	132	93.24	-523	125	26	158	87.50
0.10 OS	-538	37	80	128	94.56	-518	90	24	156	91.00
333 K										
Blank	-523	1400	150	150	—	-483	3000	75	190	—
0.02 OS	-540	507	140	144	63.79	-528	1470	73	184	51.80
0.04 OS	-535	412	142	137	70.57	-525	1146	72	180	61.80
0.06 OS	-532	277	137	128	80.21	-520	777	70	176	74.10
0.08 OS	-529	182	135	126	87.00	-513	504	68	172	83.20
0.10 OS	-527	139	134	123	90.07	-507	363	67	166	87.90

The corrosion parameters deduced from Tafel polarization such as corrosion current i_{corr} , corrosion potential E_{corr} , Tafel constant b_a and $-b_c$ and inhibition efficiency are given in Table-3. The i_{corr} values were decrease with the increasing concentration of inhibitors. The inhibition efficiencies were determined from the values of corrosion current and the inhibition efficiency values were found to show good agreement, with those obtained from mass loss measurements.

Conclusion

The conclusions reported in this paper are the following: (i) *Ocimum sanctum* acted as efficient corrosion inhibitor in 1 and 5 N phosphoric acid. (ii) The inhibition efficiencies 98.57 % in 1 N phosphoric acid and 94.64 % in 5 N phosphoric acid have been obtained with small amount of *Ocimum sanctum* (0.1 %) by mass loss technique at 302 K. (iii) Inhibition of inhibitor decreased with increasing temperature. (iv) The inhibition efficiency decreased with increasing concentration of phosphoric acid. (v) The inhibition efficiency value obtained from mass loss studies and polarization measurements showed fairly good agreement. The corrosion inhibition appears to be of mixed type. (vi) *Ocimum sanctum* inhibited corrosion by adsorption mechanism and the adsorption of these compounds from acid solution followed Temkin's adsorption isotherms at all the concentrations. (vii) Thermodynamic values obtained from this study, E_a , ΔH and ΔS indicate that the presence of the inhibitors increase activation energy and the negative values of ΔG_{ads} indicate spontaneous adsorption of the inhibitor on the surface of the mild steel. (viii) In the kinetic studies, linear plots were obtained which revealed first order kinetics.

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