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INHIBITION OF MILD STEEL CORROSION USING PLECTRANTHUS TENUIFLORUS SHARA PLANT AS SAFE AND GREEN INHIBITOR IN ACIDIC SOLUTIONS

Inhibition of Mild Steel Corrosion Using *Plectranthus Tenuiflorus (Shara)* Plant as Safe and Green Inhibitor in Acidic Solutions

Aisha M. Al-Turkustani [α], Nabeeh M. Al-Marhabi [Ω]

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I. INTRODUCTION

The meaning of the word corrosion is the substance usually a metal or its alloys which change in its properties because of a reaction with its environment. Normally it specifically applies to metals. Corrosion causes enormous losses which rise yearly with the increased using of metals industrial development. The accepted concept of the corrosion is that it is a result of an electrochemical reaction taking place on the surface of the metal where the metal is converted in to metal oxides or other corrosion products with some metals.

Corrosion affects most of industrial sector and may cost billions of dollars each year for preventing and replacement of maintenance [1]. Thus, modern world today made an investigation to overcome this problem by doing enrichment study of corrosion inhibitors. Mild steel (MS) is a material of choice and it is finds application in many industries, due to low cost and easy availability and good tensile strength besides various other desirable properties, ease to fabricate Various reaction vessels, pipes, tanks etc., in most of the chemical industries but it suffers from severe corrosion in aggressive environments [2]. Acids are used to remove oxides, shop - soil and other contaminants from metal surfaces. Acids are also used for derusting and pickling, the cleaning of refinery equipment oil well acidizing and acid descaling and the removal of calcareous deposits from boilers. Radiators of vehicles, pipelines carrying water or petroleum products heat exchangers, etc. [3].Although there are numerous options for controlling the corrosion of metals, the use of inhibitors is one of the best methods of protecting metals against corrosion.

Corrosion inhibitors are widely used in industry to reduce the corrosion rate of metals and alloys in contact with aggressive environments. Most of the corrosion inhibitors are synthetic chemicals, expensive and very hazardous to environments. Therefore, it is desirable to source for environmentally safe and green inhibitors [4,5].Natural products can be considered as a good source for this purpose. The possible replacement of some expensive chemicals as corrosion inhibitors for metals in acid cleaning process by naturally occurring substances of plant origin has been studied [6-13].

The aim of this study was to investigate the inhibition effect of *Plectranthus tenuiflorus (Shara)* plant as a cheap, safe, green, raw and non- toxic corrosion inhibitor on mild steel corrosion in sulphuric acid solution.

II. EXPERIMENTAL

a) Material

i. *Inhibitor*

Plant peraparation: *Shara* plant leaves were collected from Jeddah region; Saudi Arabia (Figure 1).

Plectlranthus tenuiflorus (Euphorbiaceae family), is the generic name for *Shara*, a perennial succulent herb; having a pleasantly aromatic juice. *Shara* her it is a large genus of the Lamiaceae family widely distributed in tropical regions of Africa, Asia and Australia [14-16] that natively grows in western and southern region of Saudi Arabia [17,18]. Several *Plectranthus* species are cultivated as ornamentals or as sources of essential oils, whereas other are used as edible tubers, or as food flavorings [15,19]. In folk medicine, they are employed for headaches, sores, burns, dermatitis, acute edematous otitis acuta, stomachache, against nausea, scorpion stings and as purgative [20-24]. In Western region, it is used as eardrop for earache and inflammation of middle ear

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[23], whereas it is prescribed in Asia for a remedy sore throat [18].

Stock solution of aqueous extract of *Shara* plant were prepared by weighing 120 g of chopped leaves mixing (blending) in a blender with 200 ml de-ionized water for 15 minuts, heating until 90° C (before boiling). The mixture is cooled for 24 hours. The aqueous extract was separated by filtration and then kept the filtrate solution in a flask at low temperatures in a refrigerator. From the stock solution of aqueous extract of *Shara* plant, the inhibitor test solutions were prepared in a concentration range (1.0%v/v – 30%v/v).

ii. *Specimen*

The metal used for the study was mild steel electrode of the chemical composition was (wt%) P (0.035), Si (0.03), Mn (0.5), Cr (0.9), C (0.38) and Fe (97.64), it was a rod with the length is 4.0 cm and diameter is 1.0 cm. The specimen surface was polished with different grade of emery papers (60, 120, 220, 400, 600, 800 and 1200), washed with de-ionized water, degreased with acetone and dried with a stream of air and weighed.

iii. *Solution*

All chemicals used were of analytic grade. The blank corroding solution was H_2SO_4 (PAI-Panreac). Appropriate concentration (2.0 M) of acid solution was prepared using de-ionized water in the absence and presence of various concentrations of aqueous extract of *Shara* Plant.

b) *Methods*

i. *Hydrogen gas evolution (Gasometric) method*

The gasometric assembly used for the measurement of hydrogen gas evolution from the reaction was as originally described [12,25]. A reaction vessel was connected to a burette through a delivery tube. The 2.0 M H_2SO_4 solution was introduced into a two-necked flask, and the initial volume of air in the burette was recorded. Thereafter, mild steel sample was dropped into the H_2SO_4 solution, and the flask quickly closed. The volume of H_2 gas evolved from the corrosion reaction was monitored by the volume change in the level of the paraffin oil. The change in volume was recorded every 5 min. for 60 min. The same experiment was repeated in the presence of the inhibitor.

Hydrogen gas evolution measurements were carried out at 30, 40, 50, 60 and 70° C. From the volume of hydrogen gas evolved per minute, corrosion rate (R), inhibition efficiency (%I) and degree of surface coverage (θ), were calculated using equations 1, 2 and 3, respectively.

$$R\ (\mathrm{ml\ cm}^{-2)} = (V^{0}{}_{Ht} - V^{1}{}_{Ht}{}') / t \qquad (1)$$

$$\%I = [1 - V^{1}{}_{Ht} / V^{0}{}_{Ht}] \times 100 \qquad (2)$$

$$\theta = \%I / 100 \qquad (3)$$

Where $V^1{}_{Ht}$ is the volume of hydrogen gas at time t for inhibited solution and $V^0{}_{Ht}$ is the volume of hydrogen gas evolved at time t for uninhibited solution.

ii. *Weight loss (Gravimetric) method*

After complete of each test mild steel sample is separated from the test solution, washed with de-ionized water and with acetone, dried with stream of air and then re-weighting. From the weight loss results, the corrosion rate (R`), inhibition efficiency (%I) of the inhibitor and degree of surface coverage (θ), were calculated using equations 4, 6 and 7, respectively,

$$R^{`}\ (\mathrm{g\ h^{-1}\ cm^{-2}}) = W/At \qquad (4)$$

$$\%I = (1 - W_1 / W_2{}^{\circ}) \times 10 \qquad (5)$$

$$\theta = 1 - W_1/W_2$$

Where W_1 and W_2 are the weight losses (g/dm^3) for mild steel in the absence and presence of the inhibitor in H_2SO_4 solution, respectively, and θ is the degree of surface coverage of the inhibition.

III. Results and Discussion

a) *Effect of inhibitor concentration*

Figure (2) shows the relation of hydrogen gas with time for mild steel corrosion in 2.0 M H_2SO_4 solution in the absence and presence of different concentrations of aqueous extract of *Shara* plant at 30° C. It is clear that, at low concentration (1.0%v/v) for the aqueous extract of *Shara* plant acceleration of the corrosion occurs (increase in the slope of the curve), and the corrosion rate decrease by increasing the concentration of the extract of *Shara* plant. Similar results were obtained from weight loss measurements. Table (1) illustrate the corrosion rates (R' and R) and inhibition efficiency (I%) obtained from WL and HE, respectively.
From Table (1), it can be seen that the values of corrosion rate for mild steel in 2.0 M H_2SO_4 solution containing aqueous extract of *Shara* plant decreased as concentration of inhibitor increased from the two methods, and the inhibition efficiency increase. This results are due to that the adsorption amount and coverage of inhibitor on mild steel surface increases by increasing the inhibitor concentration, except at low concentration (1.0%v/v) of the inhibitor which give an increase in corrosion rate (acceleration) from the two methods.

b) *Adsorption isotherm*

The nature of inhibitor interaction on the corroding surface during corrosion inhibition of metals has been deduced in terms of adsorption characteristics of the inhibitor. The decrease in the corrosion rate by the addition of aqueous extract of *Shara* plant is attributed to either adsorption of the plant components on the metal surface or, the formation of a barrier film separating the metal surface from the corrosive medium [26, 27].

Figure (3) shows the relation between I% and $\log C_{inh}$ for the investigated extract from hydrogen evolution (Gasometric) and weight loss (Gravimetric) methods. As can be readily seen, the plots have the form of *S*-shaped adsorption. This indicates that the aqueous extract of *Shara* plant inhibit the acid dissolution for mild steel by adsorption the molecules of the *Shara* plant at metal acid solution interface, also the obtained figure indicated one step of adsorption .

Basic information on the interaction between the inhibitor and the mild steel surface can be provided by the adsorption isotherm. For this purpose, the values of surface coverage (θ) at different concentrations of *Shara* plant in 2.0 M H_2SO_4 have been evaluated from weight loss and hydrogen evolution measurements using the equations (7) and (8) to explain the best isotherm to determine the adsorption process from the experimental data obtained.Attempts were made to fit these θ values to various isotherms including Frumkin, Langmuir, Temkin and Freundlich. An excellent fit was obtained for aqueous extract of *Shara* plant, using the following Langmuir adsorption isotherm equation [28].

$$\Theta = KC_{EX} / 1 + KC_{EX} \qquad (7)$$

$$C / \Theta = 1/K + C \qquad (8)$$

Where C_{EX} is the concentration of aqueous extract of *Shara* plant, K is adsorption constant and θ is degree of coverage. It can be seen from Figure (4) using equation (8) that straight lines of a slope less than unit is found. The slopes of the C/Θ versus C plots show deviation from unity, which can be attributed to the molecular interaction between the adsorbed inhibitor species on mild steel surface [29, 30].

The values of adsorption constant ($K_{ads.}$) and the standard free energy of adsorption ($\Delta G^{\circ}_{ads.}$) were calculated using the equation:

$$\ln K = \ln 1/55.5 - \Delta G^{\circ}_{ads} / RT \qquad (9)$$

Where one molecule of water is replaced by one molecule of inhibitor [31, 32]. The numerical value (1/55.5) in equation (9) stands for the molarity of water. The values of $K_{ads.}$ and $\Delta G^{\circ}_{ads.}$ for aqueous extract of *Shara plant* were recorded in Table (2). The negative $\Delta G^{\circ}_{ads.}$ values are consistent with the spontaneity of the adsorption process and the stability of the adsorbed layer on the mild steel surface [33]. It generally accepted that the values of $\Delta G^{\circ}_{ads.}$ up to -20 kJ mol^{-1} the types of adsorption were regarded as physisorption, the inhibition acts due to the electrostatic interaction between the charged molecules and the charged metal, while the values around -40 kJ mol^{-1} or smaller, were seen as chemisorptions, which is due to the charge sharing or a transfer from the inhibitor molecules to the metal surface to form covalent bond [34, 35]. The $\Delta G^{\circ}_{ads.}$ values obtained in this study equal to -46.31 to -52.89 kJ mol^{-1}. It was suggested that the adsorption mechanism of investigated inhibitor on mild steel in 2.0 M H_2SO_4 solution was typical of chemisorptions.

c) *Effect of temperature on the performance of Shara inhibitor*

To gain insight into the nature of inhibitor adsorption, the effect of temperature (30, 40, 50, 60, and 70° C) on the corrosion behavior of mild steel in the absence and presence of 5%v/v of aqueous extract of *Shara* plant was studied by weight loss and hydrogen evolution measurements. Figures (5 and 6) show the results of HE for mild steel corrosion in 2.0 M H_2SO_4 in the absence and presence of fixed concentration (5%v/v) of aqueous extract of *Shara* plant at different temperatures from 30 to 70° C. The obtained results (R' and R) and I% are given in Tables (3 and 4) and Figures (7 and 8). It is clear that, the corrosion rate of mild steel in the absence and presence of *Shara* extract increased with rising temperature, this is due to an increase in temperature usually accelerates corrosive processes, particularly in media in which H_2 gas evolution accompanies corrosion, giving rise to higher dissolution rates of the metal.The plots in Figure (8) show that the inhibition efficiency generally decreased with rising temperature apart from the observed increase in 40° C. The increase in inhibition efficiency with rising temperature can be explained may be to the synergistic effect between the molecules of the compounds occurs in *Shara* plant extract. The decrease in inhibition efficiency with increasing temperature may be attributed to a possible shift of the adsorption-desorption equilibrium towards desorption of some adsorbed inhibitor molecules from the mild steel surface due to increased solution agitation resulting from higher rates of H_2 gas evolution at higher temperatures. This shows a weak adsorption interaction between mild steel surface and the inhibitor [11, 36-39].

The apparent activation energy (E_a), enthalpy and entropy (ΔH^o and ΔS^o) for the corrosion process in the absence and presence of *Shara* extract were calculated from Arrhenius equation [40].

$$\log R = -E_a / 2.303\, RT + A \qquad (10)$$

Where A is the pre-exponential factor and *R* is universal gas constant and T is absolute temperature, and from (b) the transition-state equation

$$\log R / T = (\log R / Nh + \Delta S^o / R) - \Delta H^o / 2.303\, R \qquad (11)$$

Where *h* is plank's constant and N is Avogadro's number.

A plot of logR from ML and HE methods against1/T gives a straight lines with slope of $-E_a/2.303R$, as shown in Figure (9), E_a values for the corrosion were estimated and recorded in Table (5).

It was found that E_a value for mild steel corrosion obtained in free acid solution are of the same order of magnitude as those observed by other authors [41-43] for mild steel corrosion in some acid media. It is also of the order of activation energies encountered for the hydrogen evolution reaction. This is in accordance with the fact that the hydrogen evolution reaction in the absence of inhibitor is the rate-determining step for the overall corrosion reaction. However, the average value for the inhibited solutions is higher than that for the uninhibited solution (Table 5), indicating a strong adsorption of the inhibitor at the metal surface which leads to increase the energy barrier for the corrosion process. On the other hand, it is in the order of activation energies of diffusion processes [9, 13, 44, 45].

A plot of logR/T against 1/T (Equation 11) also gives a straight lines, as shown in Figure (10). The slopes of these lines are $-\Delta H^{\circ}/2.303R$ and the intercept is $\log R/Nh+\Delta S^{*}/R$, from which the values of ΔH° and ΔS° were deduced (Table 5). It is clear for the inhibited solution, according to Antropov and Grigoryev [46] the presence of inhibitor leads the corrosion system to pass from less random to more orderly arrangements, and hence a less negative value of entropy is observed. Values of the entropy of activation ΔS° in the absence and in presence of the studied inhibitor are negative. This implies that the activated complex in the rate determining step represents an association rather than a dissociation step [43]. This means that the activated molecules were in higher order state than that at the initial stage [47, 48].

d) Mechanism of inhibition

The obtained results indicated that aqueous extract of *Shara* plant performs a good inhibition for the corrosion of mild steel in H_2SO_4 acidic solution.

Shara plant comprises of two substances: essential oil, in which Thymol (85.3%) is the principle component. And, the whole substance leaues juice containing oxygenated trypenoids, mono terpinoid substances, seven amino acids (Ala, leu, Glu, Asp, Asn, phe and His) and several minerals including Ca, Mg and Zn [49].

The main components of *Shara* plant are hydroxyl aromatic compounds, phynolic compounds such Thymol, Tannius, Amino and Triple terbens. Such compounds facilitates the formation of a complex with the dissolved iron ions. The formation of insoluble complex on the metal surface isolates the metal from the aggressive solution and therefore inhibits the corrosion process.

IV. Conclusion

Results obtained from the two methods employed revealed that:

1. The aqueous extract of Shara plant is an effective inhibitor for mild steel corrosion in 2.0 M H2SO4.
2. The inhibition efficiency of the extract increases by increasing the extract concentration and decrease when rising temperature.
3. The inhibition efficiencies obtained by weight loss and hydrogen evolution methods are in reasonably good agreement.
4. The adsorption of the extract on mild steel surface in an acidic medium follows Langmuir adsorption isotherm.
5. The inhibition mechanism was further corroborated by the values of activation parameters obtained from the experimental data.

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Table 1 : Corrosion rates and inhibition efficiencies for mild steel in 2.0 M H_2SO_4 in the presence of different concentrations of aqueous extract of *Plectranthus tenuiflorus (Shara)* plant at 30° C.

C_{inh}(%V/V)	Corrosion Rate		Inhibition Efficiency	
	$R'_{MLM} \times 10^4$ (g. cm.$^{-2}$ min.$^{-1}$)	$R_{HEM} \times 10^2$ (ml. cm.$^{-2}$ min.$^{-1}$)	I_{MLM}%	I_{HEM}%
0.0	1.7734	7.1101	-	-
1.0	2.0091	8.9901	-13.29	-26.44
2.0	1.3553	6.0227	23.58	15.29
3.0	1.2236	5.0810	31.00	28.54
5.0	0.7785	3.2101	56.10	54.85
10.0	0.6674	2.8011	62.37	60.60
20.0	0.3955	1.6833	77.70	76.33
30.0	0.0381	0.1848	97.85	97.40

Table 2 : Adsorption isotherm parameters and correlation coefficients for *Shara* plant onto mild steel in 2.0 M H_2SO_4 at 30° C.

Methods	slope	Correlation Coefficients	$K_{ads.}$(M^{-1})	ΔG_{ads}(kJ mol^{-1})
MLM	0.9443	97.46	0.1485	- 52.89
HEM	0.9055	93.63	0.1144	- 46.31

Table 3 : Corrosion rates for mild steel in 2.0 M H_2SO_4 at different temperatures.

Temperature	30	40	50	60	70
$R'_{MLM} \times 10^4$ (g. cm.$^{-2}$ min.$^{-1}$)	1.7734	6.8532	9.9715	12.5273	23.9880
$R_{HEM} \times 10^2$ (ml. cm.$^{-2}$ min.$^{-1}$)	7.1101	35.5804	42.9553	68.2364	107.3421

Table 4 : Corrosion rates for mild steel in 2.0 M H_2SO_4 + 5%v/v of aqueous extract of *Shara* plant at different temperatures.

Temperature	30	40	50	60	70
$R'_{MLM} \times 10^4$ (g. cm.$^{-2}$ min.$^{-1}$)	0.7785	2.1023	5.0057	7.2212	20.2551
$R_{HEM} \times 10^2$ (ml. cm.$^{-2}$ min.$^{-1}$)	3.2079	11.3274	19.0520	35.8479	90.1684
I_{MLM}%	56.10	69.31	49.80	42.36	15.56
I_{HEM}%	54.88	68.16	55.65	47.47	16.00

Table 5 : Activation parameters for corrosion reaction of mild steel in 2.0 M H_2SO_4 in the absence and presence of various concentration of aqueous extract of *Shara* plant.

Method	$C_{inh.}$ (%v/v)	E_a (kJ. mol.$^{-1}$)	ΔH^* (kJ. mol.$^{-1}$)	ΔS^* (J. mol.$^{-1}$ k^{-1})
ML	0.0	50.63	47.94	-156
	5%v/v	67.07	64.39	-110
HE	0.0	53.07	50.40	-97
	5%v/v	87.74	65.06	-57

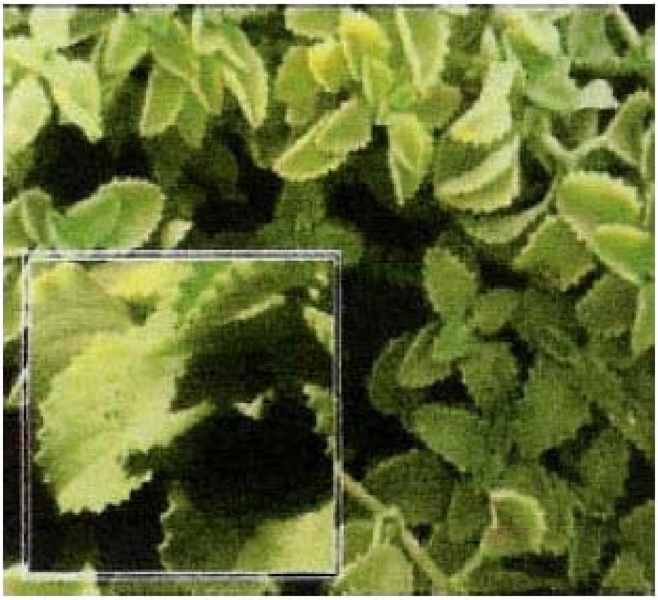

Fig.1 : Plectranthus tenuiflorus (Shara) plant leaves that collected from Jeddah region; Saudi Arabia.

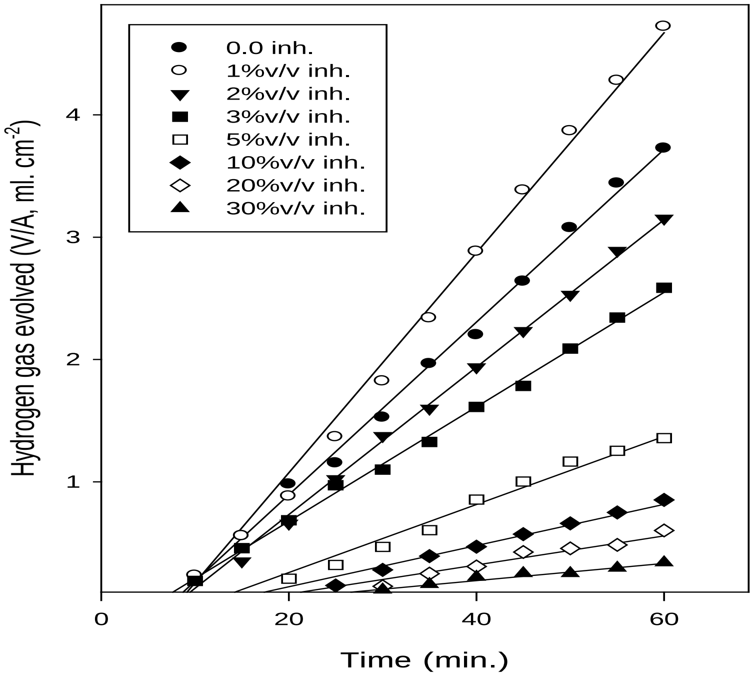

Fig.2 : Volume of hydrogen / time curves for mild steel corrosio in 2.0 M H_2SO_4 in the absence and presence of different concentratio of aqueous extract of *Shara* plant at 30 C.

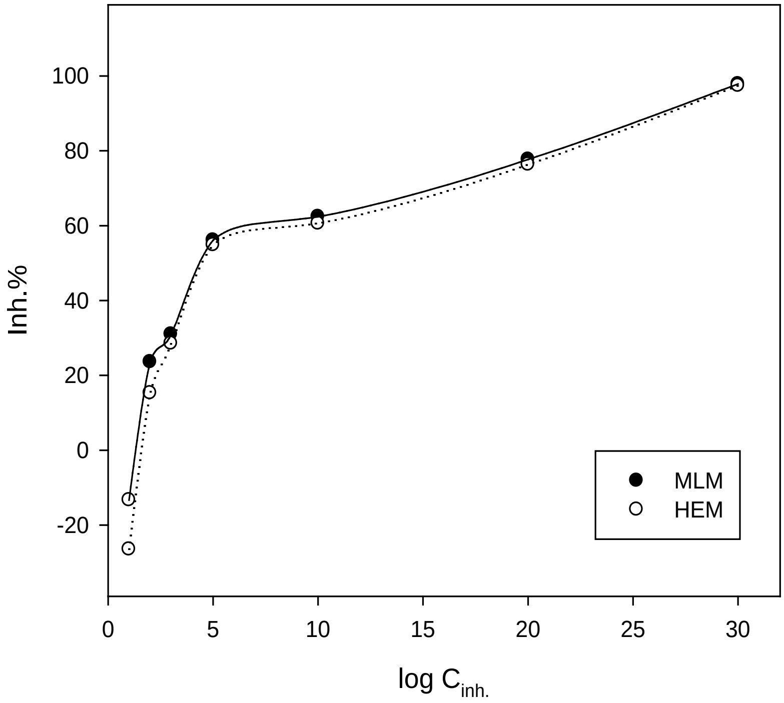

Fig.3 : The variation of inhibition efficiency (Inh.%) against log C for steel in 2.0M H_2SO_4 in the absence and presence of *Shara* extract at 30^o C.

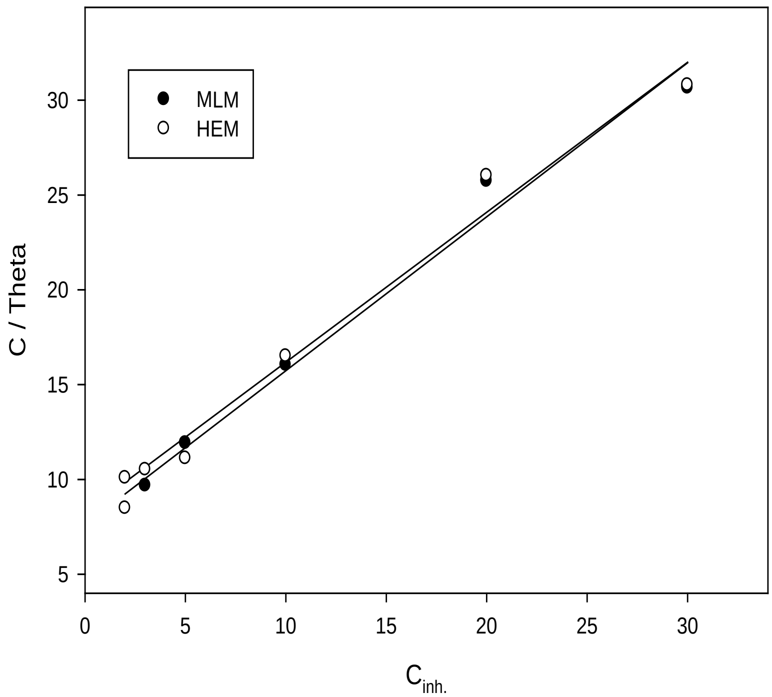

Fig.4 : The relation between C/θ against $C_{inh.}$ of aqueous extract of *Shara* for steel corrosion in 2.0 M H_2SO_4 at 30° C (Langmiur isotherm).

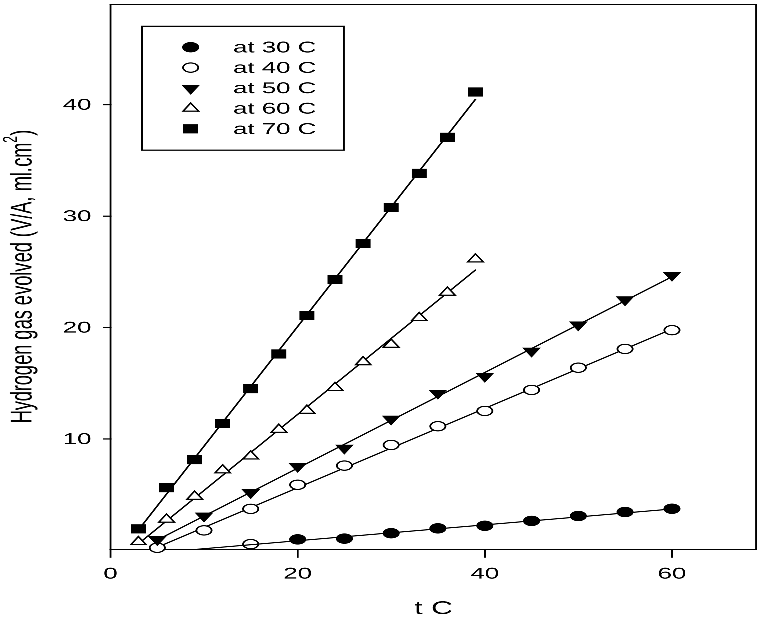

Fig .5 : volume of hydrogen/time curves for mild steel corrosion in 2.0 M H_2SO_4 at different temperatures.

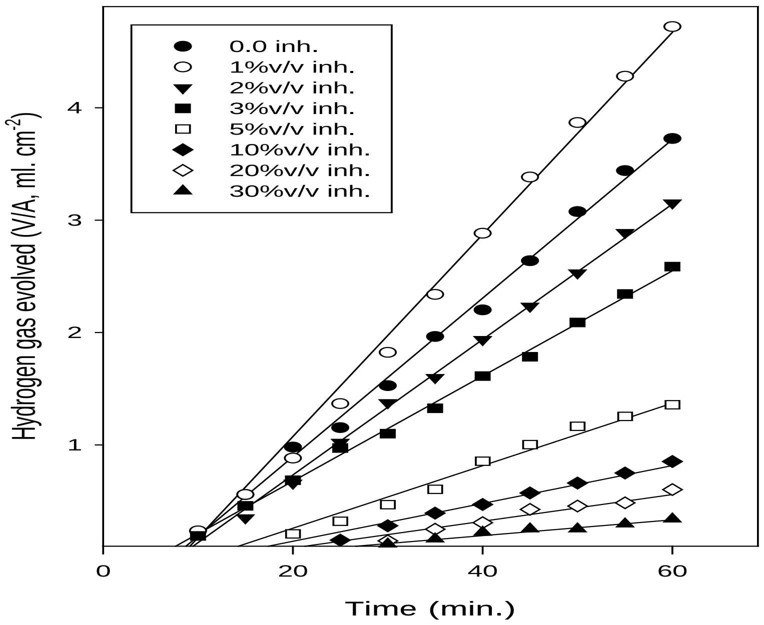

Fig. 6 : volume of hydrogen/time curves for mild steel corrosion in 2.0 M H_2SO_4 in the absence and presence of 5%v/v of aqueous extract of *Shara* plant at different temperatures.

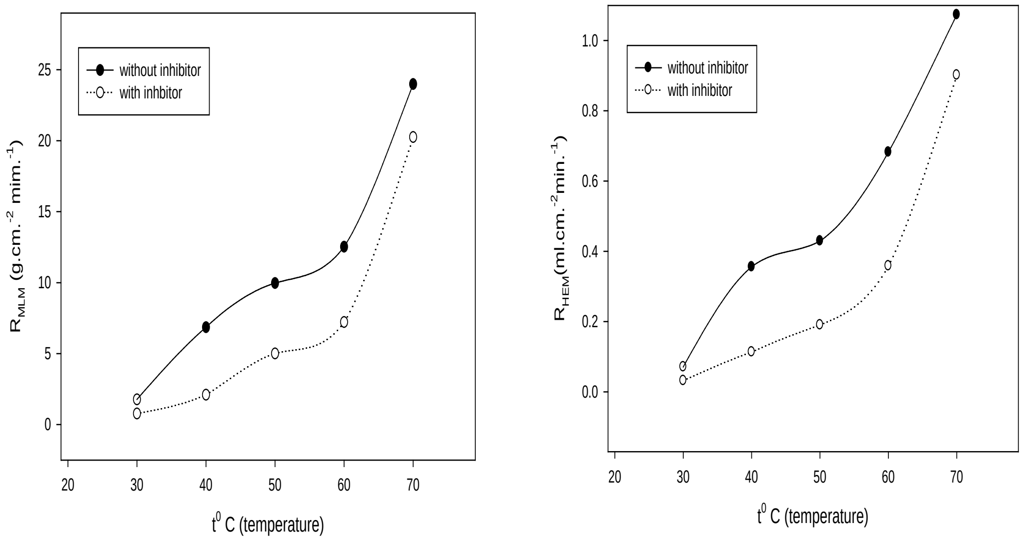

Fig.7 : The relation between corrosion rate (R_{ML} and R_{HE}) of equeous extract of *Shara* plant and temperature in H_2SO_4 solution.

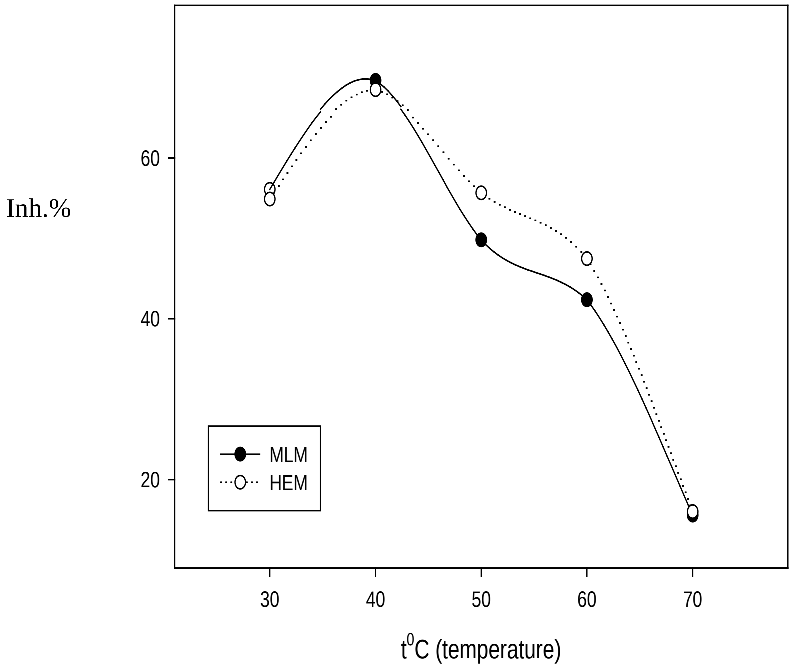

Fig.8 : The relation between inhibition efficiency (Inh.) of aqueous extract of *Shara* plant and temperature in H_2SO_4 solution.

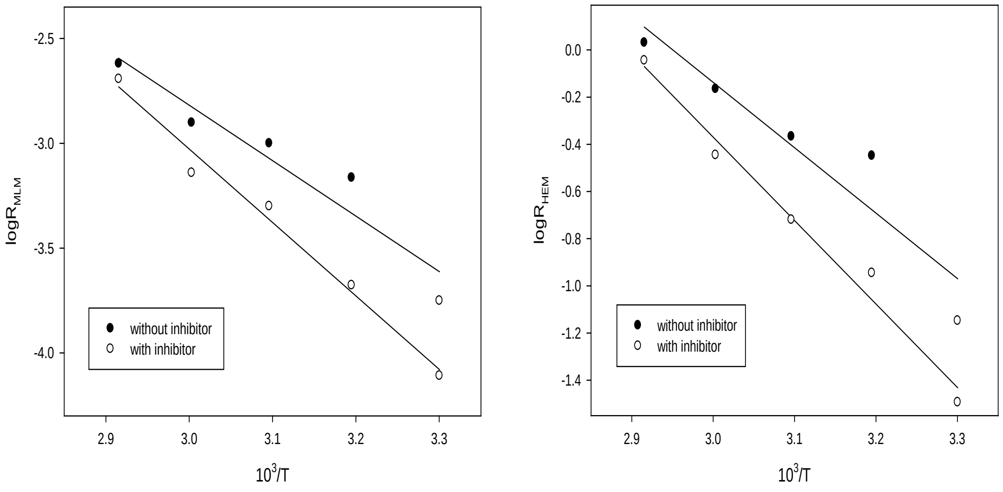

Fig.9 : Arrhenius plots for the dissolution of mild steel in 2.0M H_2SO_4 in the absence and presence of 5.0%v/v of aqueous extract of *Shara* plant.

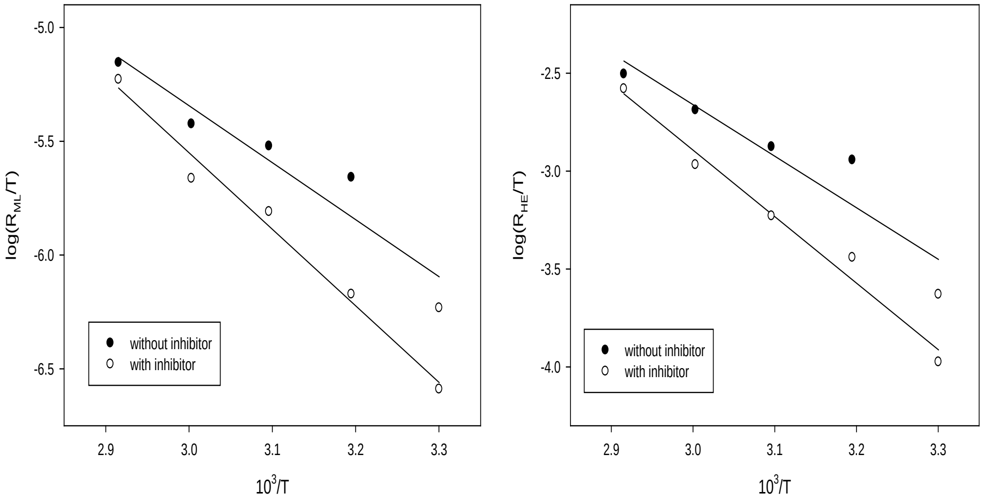

Fig.10 : The relation between log(R/T) vs. 1/T for the corrosion of mild steel in 2.0 M H_2SO_4 in the absence and presence of 5.0%v/v of aqueous extract of *Shara* plant.