ORIGINAL ARTICLE

Apricot juice as green corrosion inhibitor of mild steel in phosphoric acid

Aprael S. Yaro [a], **Anees A. Khadom** [b,*], **Rafal K. Wael** [a]

[a] *Chemical Engineering Department, College of Engineering, University of Baghdad, Aljadrea, Baghdad, Iraq*
[b] *Chemical Engineering Department, College of Engineering, University of Daiyla, Baquba, Iraq*

Abstract   The corrosion protection of mild steel in 1 M H_3PO_4 solution by apricot juice was studied at different temperatures by weight loss technique. Adsorption, activation and statistical studies were addressed in this work. Adsorption studies showed that inhibitor adsorbed on metal surface according to Langmuir isotherm. Average value of heat of adsorption was -14.93 kJ/mol indicates a spontaneous physical adsorption on metal surface. Activation parameters did not changed with addition of inhibitor indicates that there is no change in reaction mechanism. Analysis of variance (ANOVA) was also applied. This analysis showed that the corrosion rate influenced by temperature, inhibitor concentration and combined interaction of them.

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1. Introduction

Many metals and alloys which used in different human activities are susceptible to different mechanisms of corrosion due to their exposure to different corrosive media. Among these, mild steel is very important. One of the methods used to reduce the rate of metallic corrosion is addition of inhibitors. Many studies have been carried out to find suitable compounds to be used as corrosion inhibitors for this metal in different aqueous solutions.

These studies reported that there are a number of organic and inorganic compounds which can inhibit corrosion of steel [1–3]. Many researchers were conducted to examine some naturally occurring substances as corrosion inhibitors for different metals in various environments [4,5]. Naturally occurring molecules exhibiting a strong affinity for metal surfaces are the focus of research oriented toward the development of environmentally friendly corrosion inhibitors; compounds showing good inhibition efficiency and low environmental risk. Among the so-called "green corrosion inhibitors" are organic compounds that act by adsorption on the metal surface, such as ascorbic acid [6], succinic acid [7], tryptamine [8], caffeine [9] and extracts of natural substances [10–14]. The efficiency of these organic corrosion inhibitors is related to the presence of polar functional groups with S, O or N atoms in the molecule, heterocyclic compounds and pi electrons. The polar function is usually regarded as the reaction center for the establishment of the adsorption process

[15]. One of these natural compounds is fruits. Fruit is a rich source of chemicals such as vitamins, minerals, and phenolic compounds. The apricot juice includes different level of phenolic compounds. The hydroxycinnammic acids, such as caffiec, β-coumaric and ferulic acids and their esters are the most common compounds in apricot fruits [16]. Therefore, the aim of the present work is to investigate the inhibition action of apricot juice for the corrosion of mild steel in order to find a naturally occurring, cheap and environmentally safe substance that could be used for inhibition purposes.

2. Experimental work

Fully ripened apricots were purchased from a local market (Baghdad – Iraq). The fruits were washed in cold tap water and then mechanically compressed to obtain corresponding juice. The juice was then filtered to obtain a homogenous solution. In this process, one liter of apricots juice was obtained from 3 kg fresh fruit. The extracted juice was kept frozen ($> 0\,°C$) in glass bottles until further experiment. The concentrations of inhibitor were closer as 10, 20, 30, and 40 g/l. Rectangular mild steel specimens with dimensions of $3 \times 1 \times 0.1$ cm were used as the working electrodes throughout the study. The composition (wt.%) of the mild steel was: Fe, 98.9; C, 0.199; Si, 0.124; Mn, 0.053; Al, 0.0514; Cr, 0.009; Cu, 0.0468; Ti, 0.0089; V, 0.0076; Ni, 0.00352; Co, 0.0091; Mo, 0.005; Pb, 0.05, it was analyzed at Specialized Institute of Engineering Industries – Baghdad – Iraq. The cleaning procedure was as follows: The specimens were first degreased with analar benzene and acetone, and then annealed in a vacuum oven at 600 °C for 1 h and cooled to room temperature. Samples were abraded in sequence under running tap water using emery paper of grade numbers 220, 320, 400 and 600, washed with running tap water followed by distilled water, dried with clean tissue, immersed in acetone and benzene, and kept in desiccators over silica gel bed until time of use. The specimens were fully immersed for 2 h in 250 cm^3 corrosive solution at 1 M H$_3$PO$_4$, 30, 40, 50, 60 °C, and 10, 20, 30, 40 g/l apricot juice concentration. After each experiment the specimens were washed with running tap water followed by distilled water, dried with clean tissue, immersed in acetone and benzene, and kept in desiccators over silica gel bed to dry, then weighed by high accuracy electronic balance. To test reliability and reproducibility of the measurements, duplicate experiments were performed in each case of the same conditions.

3. Results and discussion

The corrosion of mild steel in 1 M H$_3$PO$_4$ solution containing various concentrations of inhibitor at different temperatures was studied by weight loss measurements. The corrosion rate of mild steel was determined using the relation:

$$W = \frac{\Delta m}{S \times t} \tag{1}$$

where Δm is the mass loss (g), S the area (m^2) and t is the immersion period (day). The percentage inhibition efficiency (IE(%)) was calculated using the relationship [17]:

$$\text{IE}\% = \frac{W_{\text{uninihibit}} - W_{\text{inhibit}}}{W_{\text{uninhibit}}} \times 100 \tag{2}$$

where $W_{\text{uninhibit}}$ and W_{inhibit} are the corrosion rates in absence and presence of inhibitor respectively. Table 1 shows the

Table 1 Corrosion rates data of mild steel in 1 M H$_3$PO$_4$ in absence and presence of different concentrations of apricots juice.

Run number	Inhibitor concentration (g/l)	Temperature (°C)	Corrosion rates (g m^{-2} day^{-1})	Inhibitor efficiency (%)
1	Nil	30	255.77	
2		40	449.3	
3		50	783.0	
4		60	1324.8	
5	10	30	96	63
6		40	210.45	53
7		50	266.24	66
8		60	508.29	62
9	20	30	83.22	68
10		40	168.96	62
11		50	243.86	69
12		60	450.1	66
13	30	30	76.73	70
14		40	155.1	66
15		50	229.48	71
16		60	410.69	69
17	40	30	64.1	75
18		40	150.63	67
19		50	211.83	73
20		60	378.14	72

variation of corrosion rates and inhibitor efficiency with inhibitor concentration and temperature. It is clear that at certain experimental temperature, corrosion rate of steel decreases with an increase in concentration of inhibitor. In absence and presence of a certain concentration of inhibitor, the corrosion rate of steel increases with rise in temperature, obeying the Arrhenius type reactions which will be discussed later. It was reported that the rate of iron corrosion in acid solutions is approximately doubles for every 10 °C rise in temperature [18]. Values of inhibitor efficiency increase with increasing inhibitor concentration.

3.1. Adsorption studies and inhibition mechanism

The primary step in the action of inhibitors in acid solution is generally agreed to be adsorption on the metal surface. This involves the assumption that the corrosion reactions are prevented from occurring over the area (or active sites) of the metal surface covered by adsorbed inhibitor species, whereas these corrosion reaction occurred normally on the inhibitor-free area [19]. Accordingly, the fraction of surface covered with inhibitor species ($\theta = \frac{\text{IE}\%}{100}$) can followed as a function of inhibitor concentration and solution temperature. The surface coverage (θ) data are very useful while discussing the adsorption characteristics. When the fraction of surface covered is determined as a function of the concentration at constant temperature, adsorption isotherm could be evaluated at equilibrium condition. The dependence of the fraction of the surface covered θ on the concentration C of the inhibitor was tested graphically by fitting it to Langmuir's isotherm, which assume that the solid surface contains a fixed number of adsorption sites and each site holds one adsorbed species. Fig. 1 show the linear plots for C/θ versus C with $R^2 = 0.9979$ correlation coefficient, suggestion that the adsorption obeys the Langmuir's isotherm:
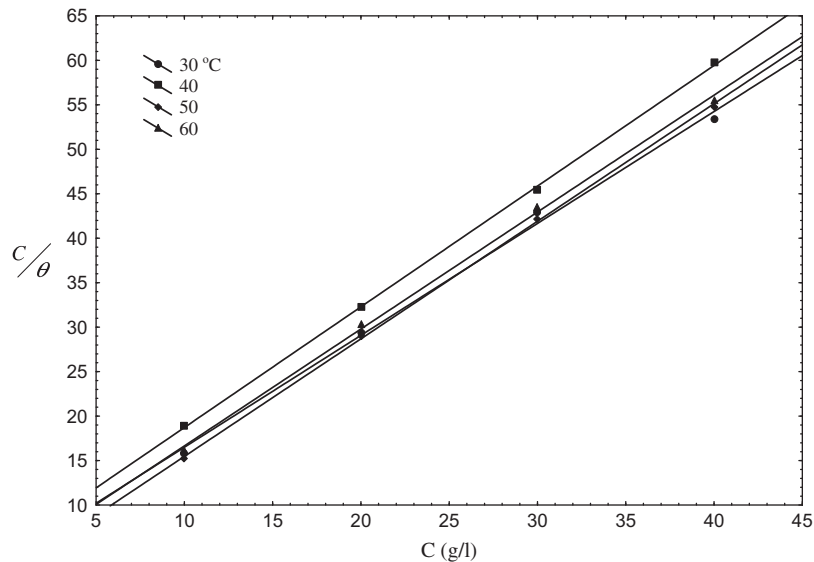
Figure 1 Langmuir adsorption isotherm for apricot juice on mild steel.

Table 2 Langmuir adsorption constants and change in free energy at different temperatures.

Temperature (°C)	K_{Langmuir} (l/g)	Slop	ΔG_{ads}^{o} (kJ/mol)	K_{Kinetic} (l/g)	y
30	0.255	1.09	−13.96	0.389	0.38
40	0.195	1.20	−13.72	0.141	0.44
50	0.446	1.10	−16.38	1.613	0.23
60	0.286	1.08	−15.66	0.438	0.32
Average value	0.296	1.11	−14.93	0.645	0.343

$$\frac{C}{\theta} = \frac{1}{K} + C \tag{3}$$

where C is the equilibrium inhibitor concentration, K adsorptive equilibrium constant, representing the degree of adsorption (i.e., the higher the value of K indicates that the inhibitor is strongly adsorbed on the metal surface), the average value of K was 0.296 l/g which obtained from the reciprocal of intercept of Langmuir plot line, and the slop of this line is near unity meaning that each inhibitor molecules occupies one active site on the metal surface.

The standard adsorption free energy (ΔG_{ads}^{o}) was calculated using the following equation [20]:

$$K = \frac{1}{999} \exp\left(-\frac{\Delta G_{ads}^{o}}{RT}\right) \tag{4}$$

where 999 are the concentration of water in solution expressed in g/l, R is gas constant, and T absolute temperature. The average value of standard adsorption free energy (ΔG_{ads}^{o}) was −14.93 kJ/mol. The negative value of ΔG_{ads}^{o} ensure the spontaneity of the adsorption process and stability of the adsorbed layer on the metal surface. Generally, value of ΔG_{ads}^{o} up to −20 kJ mol^{-1} is consistent with electrostatic interaction between the charged molecules and the charged metal (physisorption) while those around −40 kJ mol^{-1} or higher are associated with chemisorptions as a result of sharing or transfer of electrons from the molecules to the metal surface to form a coordinate type of bond [20]. While other researchers suggested that the range of ΔG_{ads}^{o} of chemical adsorption processes

for inhibitor in aqueous media lies between −21 and −42 kJ mol^{-1} [1]. Therefore, for present work the values of ΔG_{ads}^{o} has been considered within the range of physical adsorption. It was observed from Table 2, limited increase in the absolute value of ΔG_{ads}^{o} at 50 °C temperature, then, heat of adsorption decreased again at 60 °C indicating that the adsorption was somewhat favorable at this experimental temperature and apricot juice adsorbed according to physical mechanism, i.e. desorption of inhibitor molecules when temperature increased. Moreover, the essential characteristic Langmuir isotherm can be expressed in term of a dimensionless separation factor, R_L [1], which describe the type of isotherm and defined by:

$$R_L = \frac{1}{1 + KC} \tag{5}$$

The smaller R_L value indicates a highly favorable adsorption. If $R_L > 1$ unfavorable, $R_L = 1$ linear, $0 < R_L < 1$ favorable, and if $R_L = 0$ irreversible. Table 3 gives the estimated values of R_L for apricot juice at different concentrations. It was found that all R_L values are less than unity conforming that the adsorption processes is favorable.

Recent researches have looked into the action of an adsorptive inhibitor from a purely mechanistic kinetic point of view [1]. This relation can be expressed as follow:

$$\frac{\theta}{1 - \theta} = K' C^y \tag{6}$$

Table 3 The values of dimensionless separation factor R_L for apricot juice at various conditions.

Inhibitor concentration (g/l)	R_L			
	30	40	50	60 °C
10	0.281	0.339	0.183	0.259
20	0.164	0.204	0.101	0.149
30	0.116	0.146	0.069	0.104
40	0.089	0.114	0.053	0.081

or this equation can be writing in linear form as;

$$\ln\left(\frac{\theta}{1-\theta}\right) = \ln K' + y \ln C \qquad (7)$$

where K' is a constant, and y is the number of inhibitor molecules occupying one active site. A plot of $\ln(\frac{\theta}{1-\theta})$ versus $\ln C$ gives a straight line of slop y and intercept of $\ln K'$, as shown in Fig. 2. Equilibrium constant corresponding to adsorption isotherm is given by, $K = K'^{\frac{1}{y}}$. Values of $y > 1$ implies the formation of multilayer of inhibitor on the surface of metal. Values of $y < 1$ mean a given inhibitor molecules will occupy more than one active site. The behavior of equilibrium constants obtained from Langmuir model was similar to the values which obtained by kinetic–thermodynamic model. Also the values of y were lower than unity indicating the formation of monolayer on the metal surface which agree the assumptions of Langmuir adsorption isotherm.

3.2. Effect of inhibitor concentration on activation parameters

The mechanism of the inhibitor action can be deduced by comparing the apparent activation energies, E_a, in the presence and absence of the corrosion inhibitor. Activation parameters such as E_a, the enthalpy of activation, ΔH_a, and the entropy of activation, ΔS_a, for both corrosion and corrosion inhibition of mild steel in 1 M H_3PO_4 in the absence and presence of various concentrations of peach juice in the range of studied temperatures (30–60 °C) were calculated from an Arrhenius-type plot (Eq. (8)) and transition-state (Eq. (9)) [21], respectively:

$$W = A \exp\left(-\frac{E_a}{RT}\right) \qquad (8)$$

where W is the corrosion rate, A is the Arrhenius constant, E_a is the apparent activation energy, and R is the universal gas constant.

$$W = \frac{RT}{Nh} \exp\left(\frac{\Delta S_a}{R}\right) \exp\left(-\frac{\Delta H_a}{RT}\right) \qquad (9)$$

where N is Avogadro's constant, h is Planck's constant, ΔS_a is the change of entropy for activation, and ΔH_a is the change of enthalpy for activation. As shown in Figs. 3 and 4, plots of $\ln(W)$ versus $1/T$ and $\ln(W/T)$ versus $1/T$ give straight lines with slopes of $\Delta E_a/R$ and $\Delta H_a/R$, respectively. The intercepts, which can then be calculated, will be $\ln(A)$ and $[\ln(R/Nh) + (\Delta S_a/R)]$ for the Arrhenius and transition-state equations, respectively. Table 4 collects the activation parameters for mild steel corrosion reaction in H_3PO_4. The results showed positive sign for both E_a and ΔH_a, reflecting the endothermic nature of corrosion process. The values of E_a and ΔH_a varies in the same way, it is obviously seen that the activation energy increased slightly in the presence of inhibitor. This indicating that the corrosion reaction of mild steel is inhibited by apricot juice, hence supports the phenomenon of physical adsorption. The increase in the activation energy in the presence of the additives signifies physical adsorption. The negative values of ΔS_a pointed to a greater order produced during the process of activation. This can be achieved by the formation of activated complex represents association or fixation with consequent loss in the degrees of freedom of the system during the process [22].

3.3. Statistical and mathematical analysis

The experimental corrosion rate results in uninhibited and inhibited 1 M H_3PO_4 acid as a function of temperature and inhibitor concentration are given in Table 1. This table was rearranged as shown in Table 5 in matrix array. Matrix array may be helpful for statistical analysis. The statistical technique known as *analysis of variance* (ANOVA) can be used. This technique based on variance ratios to determine whether or
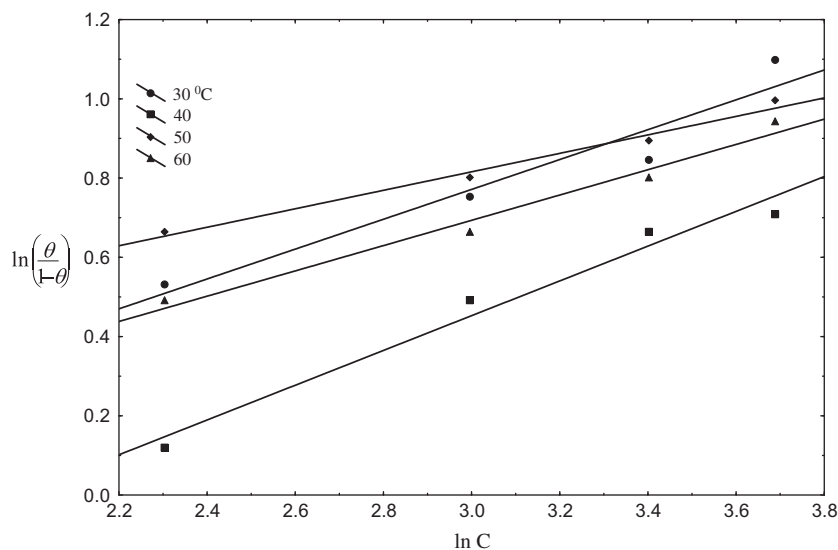

Figure 2 Kinetic–thermodynamic model for the adsorption of apricot juice on mild steel surface.
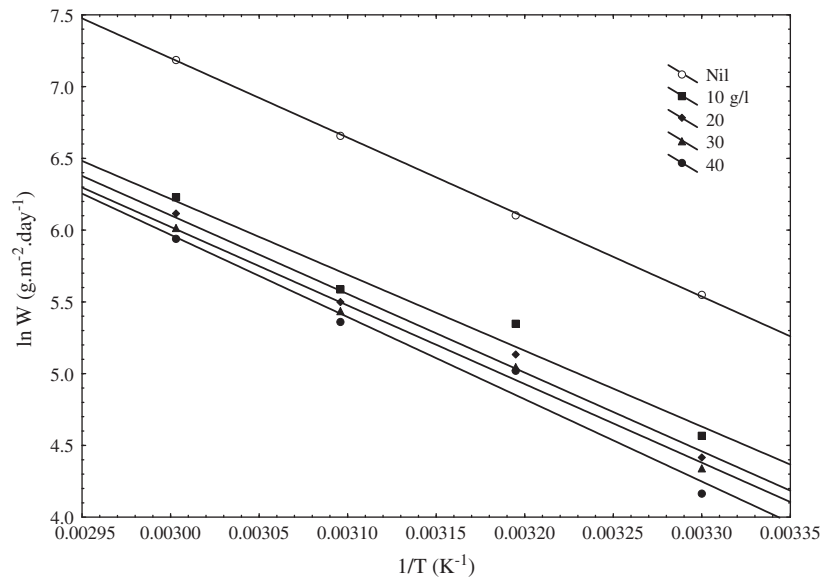
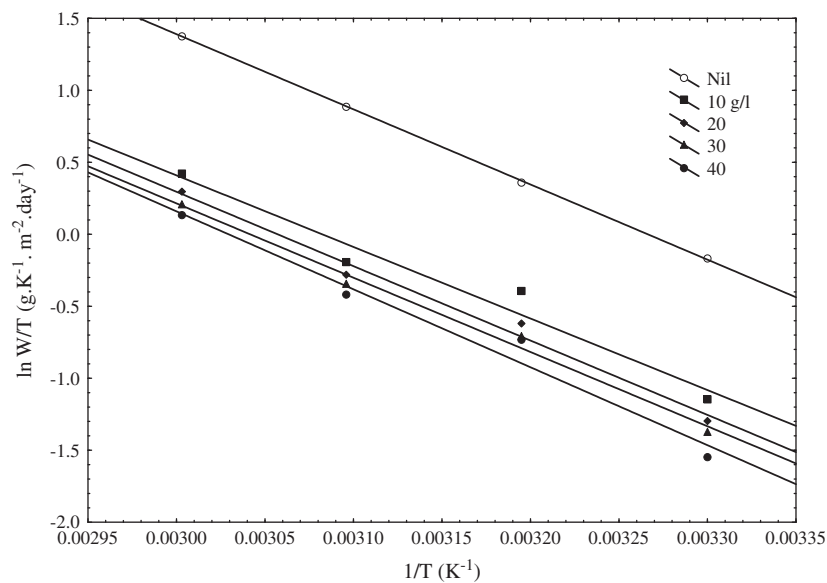**Figure 3** Arrhenius plot for the corrosion of mild steel in 1 M H_3PO_4.

Figure 4 Transition state plot for the corrosion of mild steel in 1 M H_3PO_4.

Table 4 Activation parameters for mild steel corrosion reaction in H_3PO_4.

C (g/l)	E_a (kJ/mol)	ΔH_a (kJ/mol)	ΔS_a (kJ/mol K)
0	42.02	43.38	−0.053
10	43.95	41.33	−0.071
20	45.55	42.94	−0.067
30	45.45	42.90	−0.068
40	47.56	45.00	−0.062
Average value	45.706	43.11	−0.064

not significant differences exist among the means of several groups of observation. Two-way ANOVA has been used in order to determine the effect of temperature and inhibitor concentration on corrosion rate. This will be done by determine whether variation in the column variable $X_{j\bullet\bullet}$ (temperature) affects the column means and row variable $X_{\bullet i\bullet}$ (inhibitor concentration) affects the row means. Calculation formulas were shown in Table 6. The hypothesis would be rejected if the computed F values (F), is too large. That is the rejection region is of the form [23]:

$$F > F_{K_1, K_2, (1-\alpha)} \tag{10}$$

where $K_1 = j - 1$, $K_2 = ji$ for column, $K_1 = i - 1$, $K_2 = ij$ for rows, and α is significant level (0.05). Numerical values of Table 6 were shown in Table 7. Inspection of Table 7 shows that we reject the hypothesis of no effect of column variable or the row variable. The results clearly show that temperature and inhibitor concentration at 95% confidence level respectively

Table 5 Matrix array of corrosion rate as a function of temperature and inhibitor concentration.
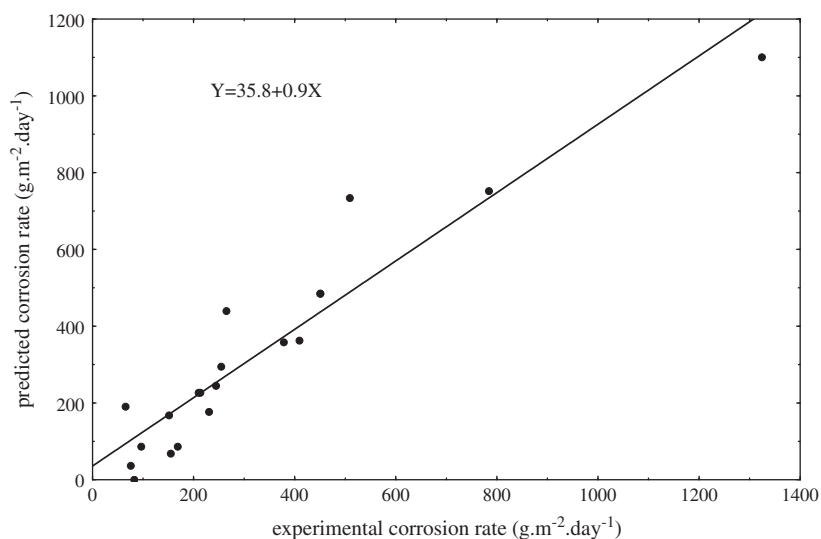
C (g/l)	Temperature (°C)				
	30	40	50	60	
0	255.77	449.3	783	1324.8	$X_{1\bullet\bullet} = 2812.87$
10	96	210.45	266.24	508.29	$X_{2\bullet\bullet} = 1080.98$
20	83.22	168.96	243.86	450.1	$X_{3\bullet\bullet} = 946.140$
30	76.73	155.1	229.48	410.69	$X_{4\bullet\bullet} = 872.000$
40	64.1	150.36	211.83	378.14	$X_{5\bullet\bullet} = 804.430$
	$X_{\bullet1\bullet} = 575.82$	$X_{\bullet2\bullet} = 1134.17$	$X_{\bullet3\bullet} = 1734.41$	$X_{\bullet4\bullet} = 3072.02$	$X_{\bullet\bullet\bullet} = 6516.40$

Table 6 Formulas of two-way ANOVA with interactions.

Source of variation	Degrees of freedom (f)	Sum of squares (SS)	Mean squares (MS)	Test statistic (F)
Between columns	$j - 1$	$SS_C = \frac{\sum_j X_{\bullet j \bullet}^2}{ik} - \frac{X_{\bullet\bullet\bullet}^2}{ijk}$	$MS_C = \frac{SS_C}{j-1}$	$F = \frac{MS_C}{MS_E}$
Between rows	$i - 1$	$SS_R = \frac{\sum_i X_{i\bullet\bullet}^2}{jk} - \frac{X_{\bullet\bullet\bullet}^2}{ijk}$	$MS_R = \frac{SS_R}{i-1}$	$F = \frac{MS_R}{MS_E}$
Interaction columns-rows	$(j-1)(i-1)$	$SS_{CR} = \frac{\sum_i\sum_j X_{ij\bullet}^2}{k} - \frac{\sum_i X_{i\bullet\bullet}^2}{jk} - \frac{\sum_j X_{\bullet j\bullet}^2}{ik} + \frac{X_{\bullet\bullet\bullet}^2}{ijk}$	$MS_{CR} = \frac{SS_{CR}}{(i-1)(j-1)}$	$F = \frac{MS_{CR}}{MS_E}$
Error	$ij(k-1)$	$SS_E = SS_T - SS_C - SS_R - SS_{CR}$	$MS_E = \frac{SS_E}{(i-1)(j-1)}$	–
Total	$ijk - 1$	$SS_T = \sum_i\sum_j\sum_k X_{ijk}^2 - \frac{X_{\bullet\bullet\bullet}^2}{ijk}$	–	–

Table 7 Numerical values of two-way ANOVA with interactions.

Source of variation	f	SS	MS	F	F_{table}
Between columns	3	929,050	309,683	3.597	$F_{3,20,0.95} = 3.1$
Between rows	4	1,076,280	269,070	3.041	$F_{4,20,0.95} = 2.87$
Interaction columns-rows	12	3,006,936	250,578	2.832	$F_{12,20,0.95} = 2.29$
Error	20	1,061,776	88,481	–	
Total	39	6,720,095	–	–	

Figure 5 Predicted corrosion rate versus experimental corrosion rate.

have a significant effect on corrosion rate. It also clear that there is an interaction between temperature and inhibitor concentration factors. According to these results, second order polynomial model was used to represent these variables. This model takes into account the effect of temperature, inhibitor concentration, and the interaction of them on corrosion rate:

$$W = B_o + B_1 T + B_2 T^2 + B_3 TC + B_4 C + B_5 C^2 \quad (11)$$

Nonlinear least squares regression analysis based on *Levenberg–Marquardt* estimation method can be used for estimation of coefficients $B_o, B_1, \ldots, B_5$, producing the following equation with 0.944 correlation coefficient:

$$W = 194.0161 - 8.2947T + 0.3895T^2 - 0.5291TC$$
$$- 10.9657C + 0.6053C^2 \tag{12}$$

where W, T and C are corrosion rate (g m^{-2} day^{-1}), temperature (°C), and inhibitor concentration (cm^3/l) respectively. Fig. 5 shows predicted corrosion rate by Eq. (12) against experimental one.

4. Conclusion

Apricot juice acts as a corrosion inhibitor of mild steel with maximum inhibition efficiency of 75% at 30 °C and maximum level of inhibitor concentration. Adsorption of inhibitor obeys Langmuir adsorption isotherm by forming a monolayer on metal surface, this result was confirmed by application of thermo-dynamic adsorption model. Values of heat of adsorption were low indicating a physical adsorption on metal surface. Statistical analysis shows that corrosion rate influenced by temperature, inhibitor concentration and the combined effect of them.

Acknowledgment

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