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Modeling and Analysis of the Corrosion Behavior of Iron in Aerated 0.1N HCl, AT Various Temperatures and Speed of Agitation using ANOVA and MATLAB

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The aim of this study is to investigate the corrosion behavior of iron in aerated hydrochloric acid at different operating conditions using multiple regression analysis based on 3^2 factorial design by generating a model to show the effect of the suggested operating conditions on the corrosion rate of iron as follows:

$$CR\ (mm/y) = 90.85 - 5.02\times10^{-3}TS + 4.08\times10^{-4}T^3 + 2.3\times10^{-7}T^2S^2 - 10^{-9}T^3S^2$$

This model was selected among three suggested models according to the highest R^2 and R^2_{adj} based on T-test with 95% confidence limit. Three speeds of agitation (stagnant, 300 and 900RPM) and three temperatures (30, 45, and 60^{o}C) were tested as independent variables in the proposed statistical model, while the corrosion rate was selected as the dependent variable. The results revealed that the model is significant according to ANOVA statistical analysis based on F and P-tests. On the other hand 3D mapping was generated using MATLAB in order to reflect the interactions among these independent variables, which showed that both independent variables might work inversely on each other, or they might work together in destructing the working electrode (iron).

Keywords: Corrosion, modeling, ANOVA, 3D mapping, hydrochloric acid, iron

1. INTRODUCTION

The application of carbon steel contributes a major role in expanding and developing industries. Iron, the major constituents of carbon steel, has proper mechanical properties that enable it to be used in constructing highway networks, bridges infrastructure, aircrafts, and so on.

Mechanical structures fabricated from iron may be erected in urban, marine, or off-shore environments, and it may face severe conditions, high temperatures, humidity, various acidic concentration (i.e. acidic rain), salinity and so on.

A multiple regression analysis was adopted in this study to build up a model that represents corrosion rate in (0.1N) aerated hydrochloric acid solution, with respect to temperature and speed of agitation.

Dissolved oxygen and hydrogen ions in acidic media will stimulate iron in order to promote material degradation [1-3].

In the light of the available information, the present work estimates the corrosion rate of iron in aerated acidic media as a function of two independent variables (speed of agitation and temperature) and their interactions by conducting 3^2 runs based on factorial design of experiment using weight loss and potentiodynamic polarization, each run was repeated for at least two times in order to ensure reproducibility of results. The results withdrawn from the applied tests in this work have been represented and correlated in a model based on ANOVA statistical method. The significance of each regressor in this model has been estimated and surface response was drawn using 3D mapping facilities in MATLAB to show the behavior of the corrosion rate of iron as a function of temperature and speed of agitation.

2. EXPERIMENTAL WORK

Two variables (i.e. temperature and speed of agitation) were studied in this work. $3^{k=2}$ factorial design of experiments was conducted with at least two representative replicas for each set of conditions.

Materials:

- Specimen

Rectangular iron coupons (99.93% Fe) of 20x10 mm exposed area were used.

- Electrolyte

The electrolyte used in this work was an aerated dilute 0.1N solution prepared from 10.7 N concentrated hydrochloric acid (A.R.). A new solution was prepared prior to each experiment using de-ionized water.

3. METHODOLOGY

Prior to each experiment the specimen was pre-weighed using sensitive balance with an accuracy of four decimal places. Afterward the rectangular specimen was exposed to aerated 0.1N hydrochloric acid for 1 hour, where the temperature and the speed of agitation were adjusted using hot plate stirrer.

This test was carried out in a jacketed standard corrosion cell of 300ml to perform the potentiodynamic polarization tests on standard flat specimens of 200 mm^2 using the Potentiostat.

Teflon-coated magnetic stirrer was used to agitate the electrolyte, and the jacketed cell was used to re-circulate hot water in order to obtain the desired temperature during each experimental run.

Once the run was accomplished, a cleaning process was conducted to remove the weakly adherent oxide layer using distilled water and paper tissue, followed by Acetone and Ethanol to ensure full removal of deposits, greases, and other contaminants, and then kept in a desiccator prior to weigh [4,5].

The loss in weight was measured and the corrosion rate was calculated (as it is shown in Table 1) at two speeds of agitation (i.e. 300 and 900RPM) as well as at stagnant condition under the effect of three temperatures of (30, 45, and 60^{o}C), furthermore Table 2 strengthen these results using second route in calculating corrosion rate according to analyzing Tafel region for each polarization curve that was drawn for each set of conditions.

Table 1. CR $_{Fe}$ obtained from weight loss measurements of iron in aerated 0.1N HCl under various temperatures and speeds of agitation

	CR$_{Fe}$ (mm/y) in 0.1N HCl								
	30^{o}C			45^{o}C			60^{o}C		
	Stagnant	300RPM	900RPM	Stagnant	300RPM	900RPM	Stagnant	300RPM	900RPM
CR $_{Fe}$ (mm/y)	96.4145	81.672	72.895	117.425	77.495	109.915	166.405	111.309	129.815

Table 2. i_{corr}, and CR$_{Fe}$ obtained from polarization curves for iron in aerated 0.1N HCl at different temperatures and speeds of agitation [figs(1-6)]

	30^{o}C			**45^{o}C**			**60^{o}C**		
	Stagnant	300RPM	900RPM	Stagnant	300RPM	900RPM	Stagnant	300RPM	900RPM
i $_{corr}$ (mA/cm^2)	7.977	7.339	6.063	10.05	7.402	8.488	16.73	8.663	10.544
CR $_{Fe}$ (mm/y)	92.5513	85.14904	70.34455	116.6028	85.87998	98.48005	194.1059	100.5104	122.3343

4. RESULTS AND DISCUSSION

Nine experiments ($3^{k=2}$) with at least two representative runs for the same condition were conducted; the average values of corrosion rate (CR $_{Fe}$) withdrawn from these experiments under several operating conditions using the weight loss measurements is shown in Table 1, while those gained from the Potentiodynamic polarization measurements were illustrated in Figs. (1-6) and Table 2.

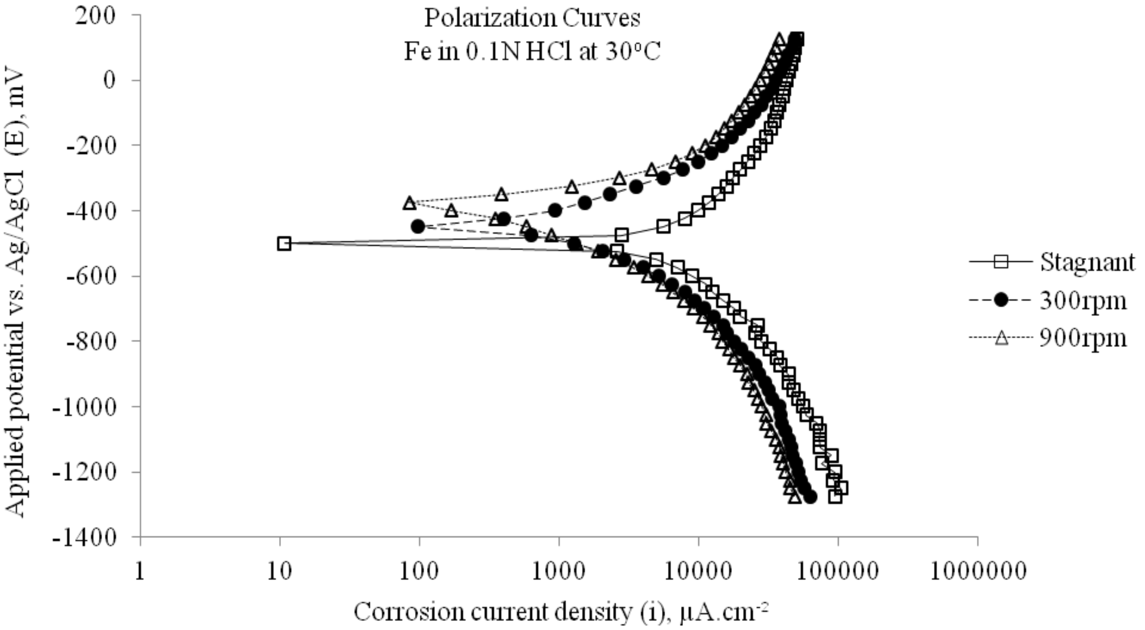

Figure 1. Polarization curves for Iron in aerated 0.1N HCl at 30°C and various speed of agitation

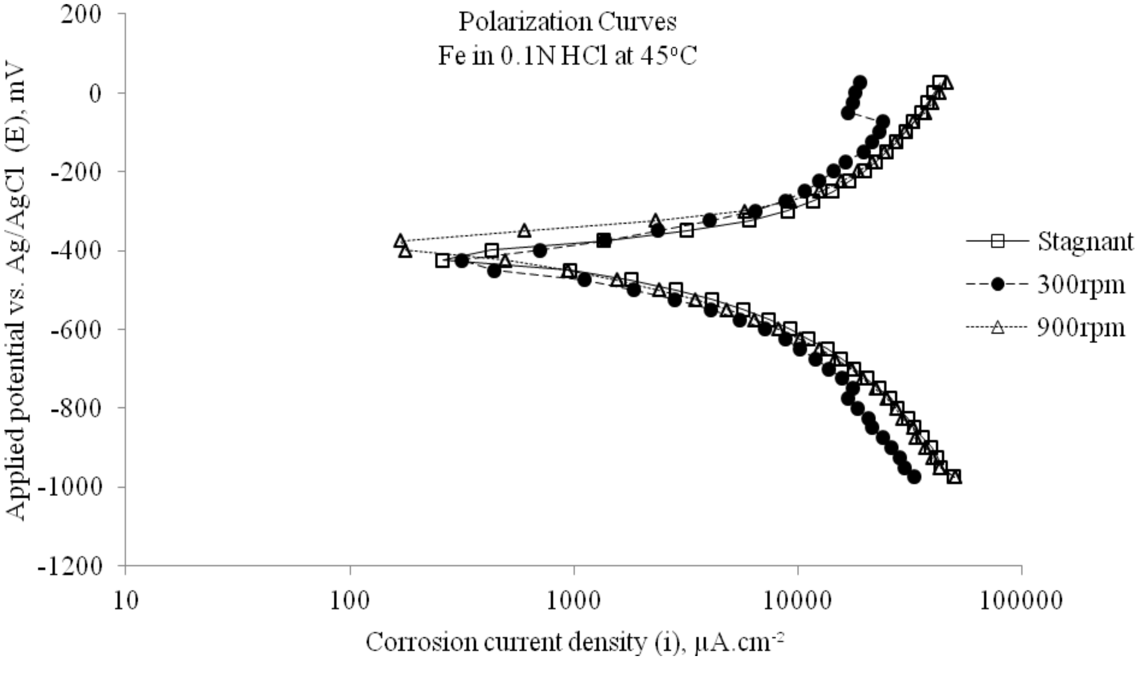

Figure 2. Polarization curves for iron in aerated 0.1N HCl at 45°C and various speed of agitation

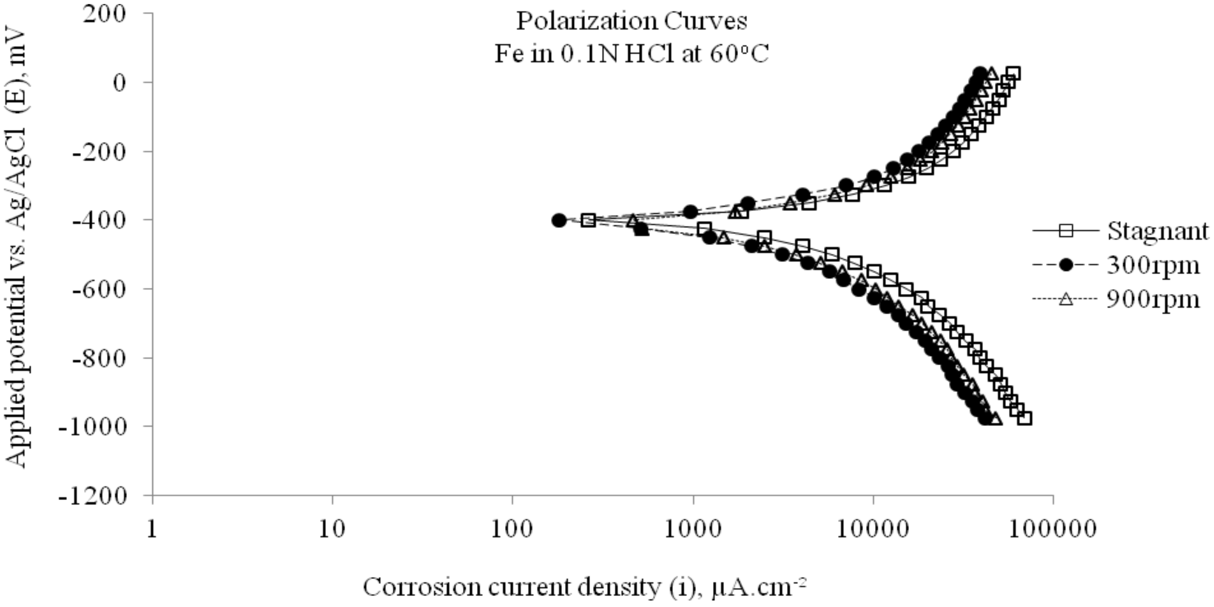

Figure 3. Polarization curves for iron in aerated 0.1N HCl at 60°C and various speed of agitation

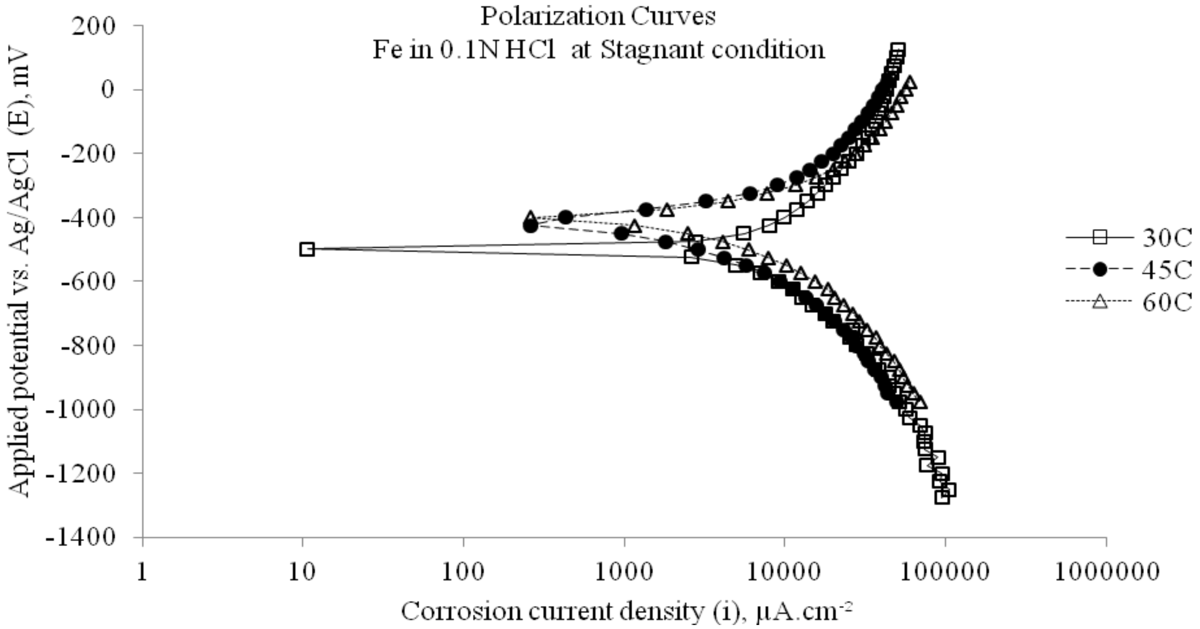

Figure 4. Polarization curves for iron in aerated 0.1N HCl at Stagnant condition and various temperatures

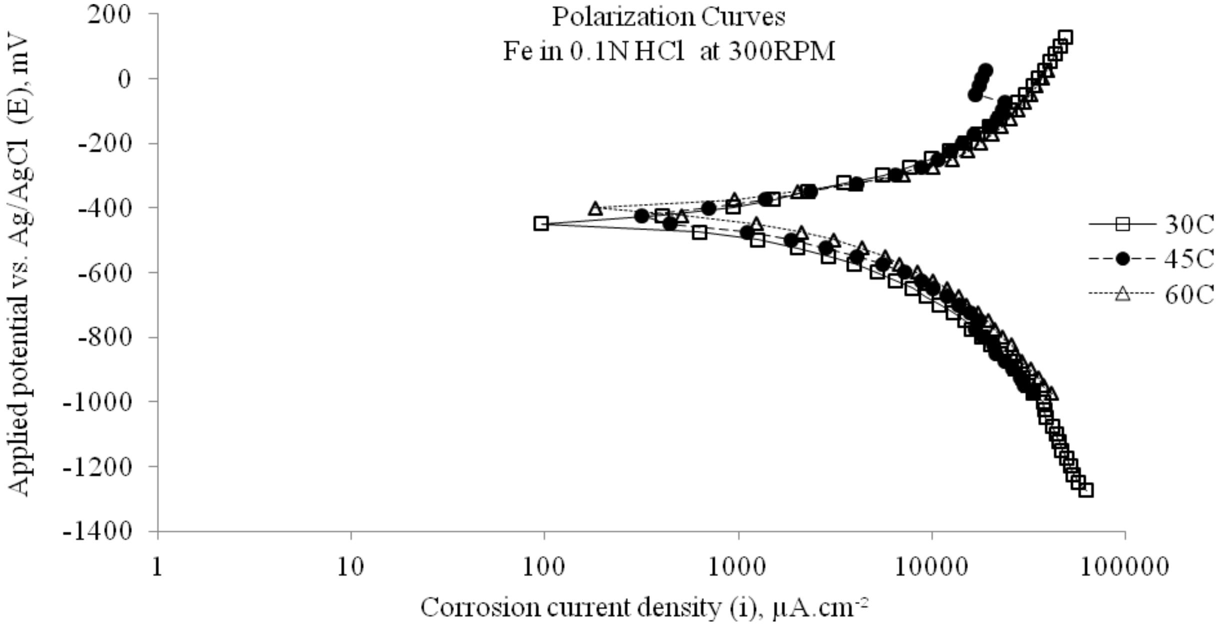

Figure 5. Polarization curves for iron in aerated 0.1N HCl at 300RPM and various temperatures

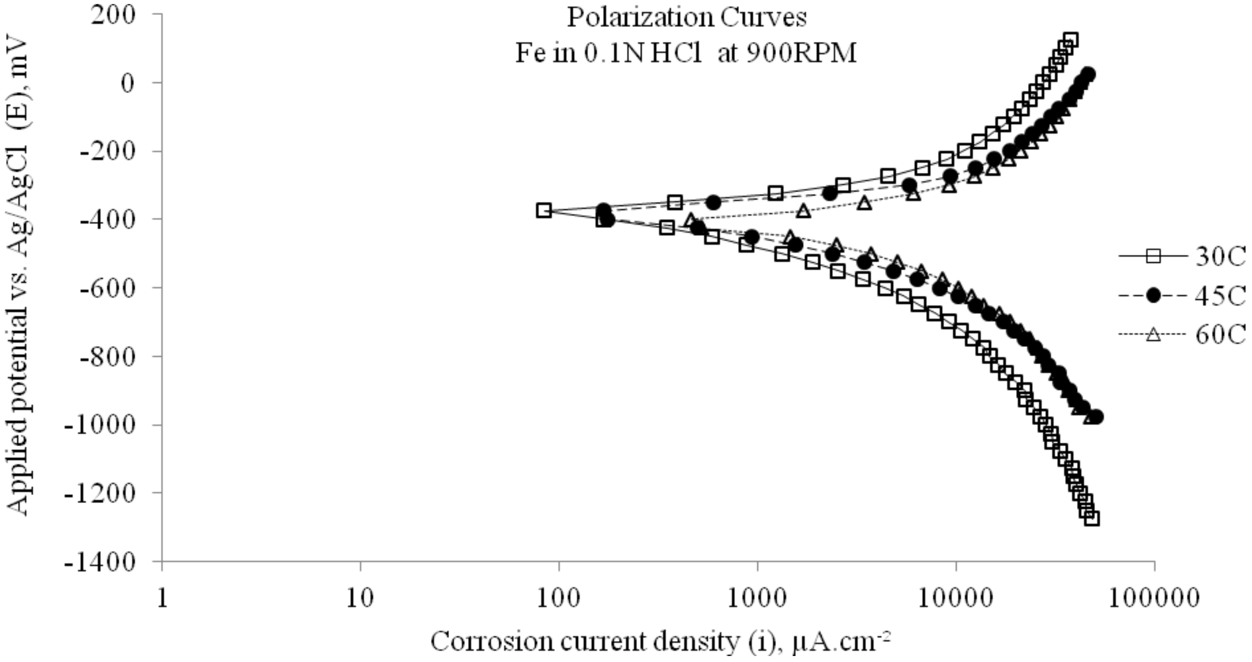

Figure 6. Polarization curves for iron in aerated 0.1N HCl at 900RPM and various temperatures

4.1. Effect of speed of agitation

Tables (1&2) shows that CR_{Fe} at fixed temperatures changes (increases or decreases) with increasing the speed of agitation [6]. The speed of agitation will play as an erosion factor in destructing the oxide film and as a result the corrosion rate will be increased. In other word the thickness of the oxide film would be reduced with respect to time while increasing the speed of agitation together with reducing the distance and the time needed for a diffused ion to be adsorbed at the to metal surface [7,8]. Badiea and Mohana stated that, increasing the speed of agitation damage the formed oxide layer, as a result oxygen ions will easily reach the metal surface causing an increase in corrosion [9]. This case happened at 45 and 60°C when the speed of agitation is increased from 300RPM up to 900 RPM as shown in Tables (1&2).

On the other hand CR_{Fe} might be slightly decreased with increasing the speed of agitation. This case occurred when the speed of agitation was increased from 0 to 900RPM at 30°C, and it was also noticed at 45, and 60°C when speed was changed from the stagnant condition to 300RPM as shown in Tables (1&2), which means that speed of agitation thickened the oxide film layer along the metal surface which will not permit any further ions to diffuse this film and as a result protects metal from any further attack. This behavior of iron is compatible with the explanation of Kim, *et. al.* [6], Shifler [7] , and Paul *et. al.* [8].

4.2. Effect of temperature

The relationship of the corrosion rate of iron given in tables 1 and 2, as a function of temperature was illustrated in Fig. 7 which summarizes the dependence of corrosion rate on temperature.

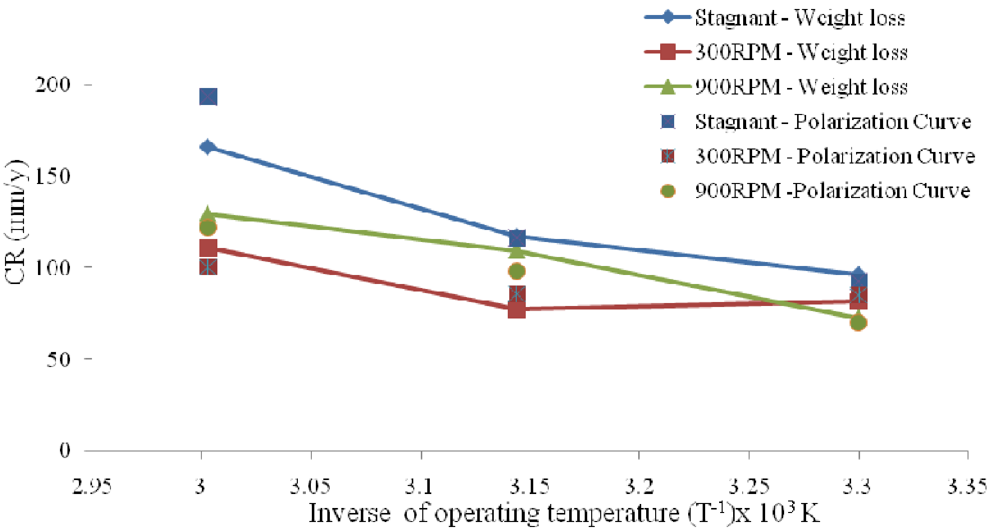

Figure 7. Arrhenius plots of iron in aerated 0.1N HCl at stagnant condition as well as in the presence of 300, and 900 RPM

Raising the temperature from 30^{o}C (303K) up to 60^{o}C (333K), increased the corrosion rate of iron in both tests (weight loss and potentiodynamic polarization) for each speed of agitation, the explanation of this behavior is compatible with the explanation given by Shifler [7].

4.3. Model generation and analysis

Empirical correlations were generated based on T tests with 95% confidence limit as shown in Table 3. A polynomial with a fifth degree was chosen with the highest R^2 and R^2_{adj} among the other two suggested models as follows (i.e. model no.1 in Table 3):

$$CR(mm/y) = 90.85 - 5.02x10^{-3}TS + 4.08x10^{-4}T^{3} + 2.3x10^{-7}T^{2}S^{2} - 10^{-9}T^{3}S^{2} \qquad (1)$$

where T is temperature in (^{o}C), and S is speed of agitation in (RPM), with a correlation coefficient of (R^2=89.8%), and R^2_{adj}=79.6% [10].

Table 3. Estimation of regression coefficient for different suggested models base on T-test with 95% confidence limit

	1st Model	2nd Model	3rd Model
Predictor	Coefficient	Coefficient	Coefficient
Constant	90.85	91.28	34.8
T	n/a	n/a	1.834
T^2	n/a	n/a	0.00821
TS	-0.005023	-.004957	-0.004870
T^3	0.00040816	0.0004035	n/a
T^2S^2	0.00000023	0.00000014	0.00000004
S^2	n/a	-0.00001864	0.00010688
T^3S^2	-0.00000000	0.00000026	n/a
R^2	0.898	0.898	0.846
R^2_{adj}	0.796	0.731	0.59

Table 4. ANOVA table of the adopted model (polynomial of the fifth degree)

Source	DF (Degree of Freedom)	SS (Sum Square of error)	MS (Mean Square of error)	F -test	P - test
Regression	4	8822.7	2205.7	8.78	0.029
Residual error	4	10004.4	251.1		
Total	8	9827			

Table 4 shows the analysis of variance of the selected model (equ. 1) which emphasize that the model is significant, since $F_{o,\ table\ 4}$ (= 8.78) > $F_{0.05,4,4}$ (= 6.39) as it is tabulated in F-tables, and consequently P test is also valid ($P_{o,\ table\ 4}$=0.029) < (α=1-confiedence limit = 0.05) [11]. The numerical estimates of the regression coefficients given in (equ.1) are shown in Table 5.On the other hand Fig. 8 shows the surface response of the selected model (equ. 1). The surface is considered to be concaved up with a lowest value of corrosion rate (72.875 mm/y) at 30°C and 900RPM, followed by a corrosion rate of (77.495 mm/y) at 45°C and 300RPM. The highest corrosion rate occurred at 60°C, stagnant condition. This 3D graph summarizes the effect of both independent variables (speed of agitation and temperature) on corrosion rate and easily revealed the interaction that occurred between these independent variables since the surface was not smooth one [12].

Table 5. Numerical estimates of the regression coefficients

Predictor	Coefficient	SE Coefficient (Standard Error)	T	P
Constant	90.85	12.32	7.38	0.002
TS	-0.005023	0.001287	-3.9	0.018
T^3	0.00040816	0.00008754	4.66	0.01
T^2S^2	0.00000023	0.00000007	3.39	0.028
T^3S^2	-0.00000000	0.00000000	-2.92	0.043

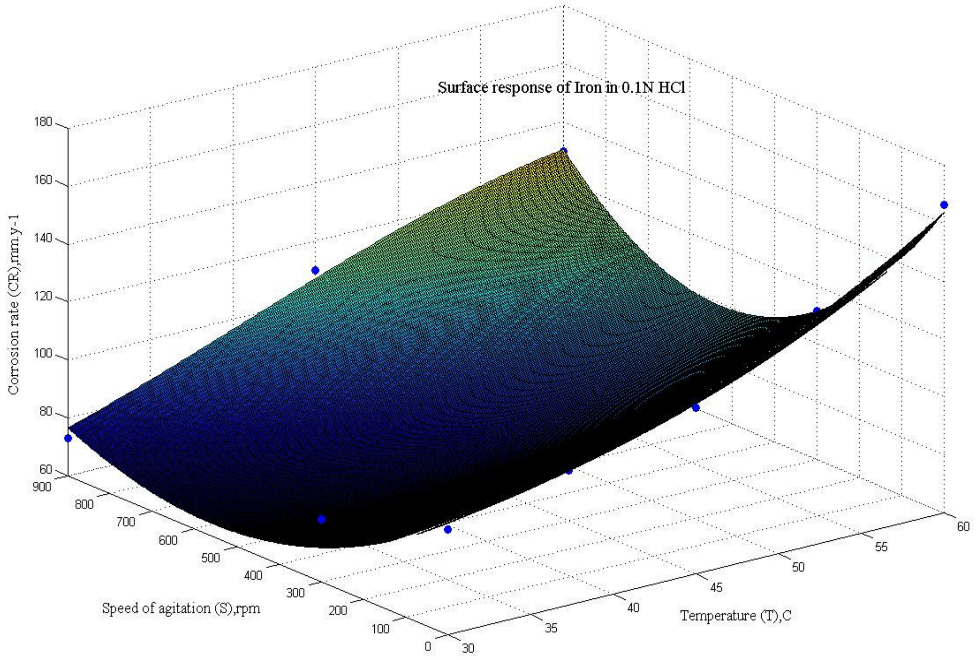

Figure 8. Surface response of the corrosion rate of iron in aerated 0.1N HCl under the effect of speed of agitation and temperature

5. CONCLUSIONS

1. The following model was selected among two other models based on the highest R^2 and R^2_{adj} , to show the effect of suggested operating conditions as follows:
   $CR\ (mm/y) = 90.85-5.02x10^{-3}TS+4.08x10^{-4}T^3+2.3x10^{-7}T^2S^2-10^{-9}T^3S^2$
   $R^2 = 89.9\%$ and $R^2_{adj} = 79.6\%$.
2. ANOVA statistical analysis showed that the selected model is significant based on F and P-tests.
3. The surface response of the selected model using 3D mapping facilities in MATLAB showed obvious interactions among the independent variables (speed of agitation and temperature) which might work inversely on each other by reducing the rate of corrosion, or they might work together in increasing the rate of corrosion and in destructing the working electrode (iron).

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