Statistical analysis and optimization of the corrosion inhibition efficiency of a locally made corrosion inhibitor under different operating variables using RSM

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Abstract

Corrosive solutions such as hydrochloric acid find huge applications in many manufacturing processes such as pickling, cleaning and acid removal techniques due to effective cleaning procedures and low costs. The utilization of corrosion inhibitors is a significant practical technique to reduce the corrosion of mild steel in corrosive solutions. Organic molecules with hetero atoms such as sulfur, phosphorous, oxygen, and nitrogen are the best corrosion inhibitors. 4-Amino-3-(2-bromo-5-methoxyphenyl)-1*H*-1,2,4-triazole-5(4*H*)-thione (ATH) was synthesized by cyclization of 2-bromo-5-methoxybenzohydrazide in the presence of carbon disulfide and hydrazine. ATH was characterized by proton, carbon-13 nuclear magnetic resonance (NMR) and Fourier transform infrared (FT-IR) spectroscopy techniques in addition to carbon, hydrogen and nitrogen elemental analysis (CHN elemental analysis). ATH was investigated as a sustainable inhibitor for mild steel corrosion in acidic medium using corrosion experiments. Response surface method (RSM) was used in obtaining the optimum operating conditions, interactive and main effect of the parameters inhibiting the mild steel corrosion. The coefficient of determination (R^2) and ANOVA (analysis of variance) proved the RSM method appropriate for the optimization of waste product inhibition on mild steel. The predicted and experimental values from the model are in good agreement. This study suggests that (ATH) is a promising significant corrosion inhibitor.

Keywords: *mild steel corrosion, response surface method, optimization, 4-amino-3-(2-bromo-5-methoxyphenyl)-1H-1,2,4-triazole-5(4H)-thione.*

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Introduction

Corrosion is an important and critical issue faced in various industries. The offshore structures exposed to chloride solutions, rain, condensation, and water, constantly lead to severe deterioration. The pitting and localized corrosion are caused by chloride ion which de-passivate the metal and makes them susceptible to deterioration at certain threshold chloride concentrations [1–3].

Among various methods of preventing mild steel corrosion in chloride solution, the use of inhibitor has been considered the best method owing to its practical technique and low cost. Inhibitor efficiency depends on the nature of the metal surface, environment and inhibitor structure [4, 5].

Organic compounds having hetero-atoms such as S, N and O have been reported by different researchers as efficient inhibitor for various metals [6–10].

The common inhibitors that were being used are nitrates, benzoates, phosphates, and chromates. In recent times, the eco-friendly corrosion inhibitor is been used to control metallic corrosion to replace the toxic, expensive and scarce corrosion inhibitors. Experiments are useful in explaining the corrosion inhibition mechanism, however, they are time-consuming because it is constantly based on large-scale trial and error experiments and also expensive.

RSM is used widely in different engineering fields to optimize the engineering process, estimate the individual factor effect and their interactive effect. Therefore, RSM could be applied in inhibition of mild steel corrosion due to its ability to reduce process duration, process variables, and overall cost. Hence, the aim of this study was to identify the optimal conditions for (ATH) inhibition and examine the effects of concentration, temperature and exposure time on (ATH) inhibition using the RSM approach. Microscopic examination of the mild steel surface after weight loss tests was also discussed in this study.

Experimental work

Chemistry and synthesis of the corrosion inhibitor

Solvents and starting materials were purchased from Sigma Aldrich Malaysia and have been utilized as they are. The melting point was measured (uncorrected) utilizing the standard melting point device. FT-IR spectrum was performed on 8300 spectrometer/Shimadzu. CHN analyses have been done through using of 5500 CHN elemental analyzer/Carlo Erba. The NMR spectrum was recorded in at 300 MHz/Bruker Spectrospin with internal standard namely tetramethylsilane.

Bromo-5-methoxybenzohydrazide was synthesized by refluxed an ethanolic solution of equimolar quantities of methyl 2-bromo-5-methoxybenzoate (0.03 mol) and hydrazine hydrate (0.03 mol) for 5 h.

The thin-layer chromatography technique was used to monitor the completion of the reaction. The reaction mixture was cooled, then filtered, dried and recrystallized from ethanol. Yield 83%, FT-IR (ν max cm^{-1}): 3348.5 and 3219.1 for amino groups; 3077.5

(Aromatic C–H); 2877.8 (–OCH_3), 1651.3 (C=O stretching), 766.2 (C–Br). ^{1}H NMR in DMSO-d_6: δ (ppm) 3.78 (2H, s, NH_2), 3.53 (3H, s, –CH_3), 7.21–7.40 (H, m, Ar–H), 8.93 (1H, s, NH). ^{13}C NMR in DMSO-d_6: δ (ppm) 114.1, 119.2, 122.9, 134.4, 139.5 and 143.6 for (carbon benzene ring), 55.2 (methyl) and 161.7 for carbonyl group. CHN Analysis. Calculated/found for $C_8H_9BrN_2O_2$: C, 39.21/38.99, H, 3.70/3.57, N, 11.43/11.81.

4-Amino-3-(2-bromo-5-methoxyphenyl)-1*H*-1,2,4-triazole-5(4*H*)-thione accordingly: carbon disulfide (0.039 mol) was added to a mixture of 2-bromo-5-methoxybenzohydrazide (0.03 mol) with absolute ethanol (50 ml) and potassium hydroxide (4.6 g). The mixture was stirred for 10 hours at room temperature. Then diethyl ether (30 mL) was added to the mixture with shaking for one hour. The salt was separated, filtered and washed with ether (15 mL). Hydrazine hydrate (0.06 mol) was added gradually to a mixture of potassium salt (0.03 mol) and water (36 ml) then refluxed for 3 hours. The H_2S gas evolved and the color changed to dark green. The resulting mixture was cooled to 5°C and then acidified by hydrochloric acid to pH 1.00. The mixture was filtered and the product was washed with distilled water and recrystallized from ethanol. IR (KBr) γ/cm^{-1}: 3171.36–3300.57 (NH_2 stretching), 3071.36 (aromatic CH stretching), 2939.95 (methyl CH stretching), 1611.23 (C=N stretching), 1493.11 (C=S), 746.93 (C–Br); ^{1}H NMR: (DMSO-d_6): δ 3.78 (s, 3H, –OCH_3), 6.17 (dd, 1H, *J* = 8.6, 2.8 Hz for –NH_2), 7.34–7.38 (dd, 2H, *J* = 2.8, 0.5 Hz, ArH), 7.58–7.79 (dd, 1H, *J* = 8.4 Hz, ArH); ^{13}C NMR (DMSO-d_6): δ 55.97, 112.31, 116.11, 120.91, 127.04, 133.51, 147.22, 157.11, 170.31; CHN Analysis. Calculated/found for $C_9H_9BrN_4OS$: C, 35.89/36.22; H, 3.01/3.08; N, 18.60/18.67.

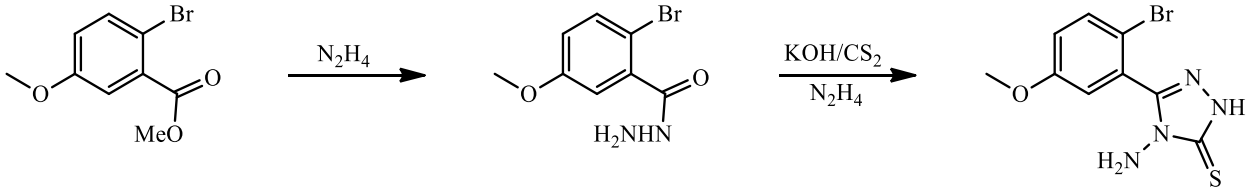

Figure 1. The synthesis of the corrosion inhibitor.

Corrosion test

Coupons of mild steel with dimension s of 2.5 cm × 2.0 cm × 0.05 cm with the composition (wt. %): 99.21% Fe; 0.21% C; 0.38% Si; 0.09% P; 0.05% S; 0.05% Mn and 0.01% Al were used for weight loss and surface analysis studies.

The mild steel coupons were mechanically polished utilizing various grades of emery papers, then the use of acetone for degreased and then washed with distilled water, dried and stored for use. A solution of HCl (hydrochloric acid 37% analytical grade) was diluted with distilled water for the purpose of preparing a 1 M acid solution with and tests were performed at 303 K.

Gravimetric study

Weight loss tests were performed by immersion in the mild steel coupons in 100 mL of the HCl environment of 1 molar, in the presence of corrosion inhibitor in concentrations of 0.0, 0.01, 0.02, 0.03, 0.04 and 0.05 at 303 K.

After immersion of mild steel coupons for 5 hours, the coupons were washed with distilled water, acetone, dried, and re-weighed to calculate weight loss, using the ASTM procedure [11, 12]. These experiments were repeated two times, using average weight loss values to calculate the corrosion rate (C_R), ($mg \cdot cm^{-2} \cdot h^{-1}$) according to equation 1 [10, 13].

$$C_R = \frac{w}{At} \tag{1}$$

Where w is the weight loss (mg), A is the area of mild steel coupon (cm^{-2}), and t is the immersion time (h^{-1}). The inhibition efficiencies and surface coverages can be calculated according to equations 2 and 3 respectively [14].

$$IE(\%) = \frac{C_R - C_{Ri}}{C_R} \tag{2}$$

$$\theta = \frac{C_R - C_{Ri}}{C_R} \tag{3}$$

Where C_R is the corrosion rate in the absence of the tested inhibitor, and C_{Ri} is the corrosion rate in the presence of the tested inhibitor.

Surface morphology analysis

Morphology of the MS surface that was immersed in a corrosive solution with/without the inhibitor was done in this method. The concentration used in this test was 1000 g/L at room temperature of 303 K for 5 hours. Surface analysis was done using the SEM technique.

Design of experiments using response surface method

The response surface method is a set of statistical and mathematical techniques to model and analyzes problems where the response of interest is influenced by different variables [15]. This method was adopted due to its ability to estimate interactive effect between tested parameters, and its limiting ability of actual experimental number to be carried out compared to other methods.

The key parameters influencing inhibitor performance were believed to be exposure time, inhibitor concentration and temperature [16].

Response Surface Methodology (RSM) model at different levels for three variables was used as the experimental design model. More than 100 readings were used in calculating the polynomial equation coefficients fitted to the experimental data.

In this study, (ATH) concentration, temperature and exposure time were the independent variables. After the determination of parameters, a mathematical model was

used to plot a three-dimensional graphics for all reaction factors and optimal reaction conditions were determined to obtain the optimum operating condition for the inhibitor.

In addition, analysis of variance was utilized to establish the relationship between experimental and predicted values using statistical parameters [15]. The model terms are tested by Fisher's exact test (*F* value) and significance probability (*P*-value).

Shown in Table 1 is the experimental range, independent variables, and design model levels. The actual matrix experimental design is shown in Table 2.

In developing corrosion process regression equations, the third-order polynomial equation was used. The main interactive effect of the possible factor combinations has been estimated. The proposed equation for which the coefficients were sought to be found was:

$$Y = \beta_0 + \beta_1 \cdot x_1 + \beta_2 \cdot x_2 + \beta_3 \cdot x_3 + \beta_4 \cdot x_1^2 + \beta_5 \cdot x_2^2 + \beta_6 \cdot x_3^2 + \beta_7 \cdot x_1 x_2 + \beta_7 \cdot x_1 x_2 + \beta_8 \cdot x_1 x_3 + \beta_9 \cdot x_2 x_3 \quad (4)$$

Where x_1, x_2, and x_3 are the concentration, time and temperature respectively.

Table 1. Experimental range and variable levels.

Variable	Low-Level Factor	High-Level Factor
Inhibitor Concentration (mg)	–1 (0 mg)	+1 (5 mg)
Temperature (°C)	–1 (30°C)	+1 (60°C)
Exposure Time (hr)	–1 (1 hr)	+1 (48 hr)

Table 2. Experimental design for RSM model design in terms of actual variables.

StdOrder	RunOrder	Blocks	PtType	Concentration	Time	Temperature
1	1	1	1	0.0	1	303
2	2	1	1	0.1	1	303
3	3	1	1	0.2	1	303
4	4	1	1	0.3	1	303
5	5	1	1	0.4	1	303
6	6	1	1	0.5	1	303
7	7	1	1	0.0	1	313
8	8	1	1	0.1	1	313
9	9	1	1	0.2	1	313
10	10	1	1	0.3	1	313
11	11	1	1	0.4	1	313
12	12	1	1	0.5	1	313
13	13	1	1	0.0	1	323

StdOrder	RunOrder	Blocks	PtType	Concentration	Time	Temperature
14	14	1	1	0.1	1	323
15	15	1	1	0.2	1	323
16	16	1	1	0.3	1	323
17	17	1	1	0.4	1	323
18	18	1	1	0.5	1	323
19	19	1	1	0.0	1	333
20	20	1	1	0.1	1	333
21	21	1	1	0.2	1	333
22	22	1	1	0.3	1	333
23	23	1	1	0.4	1	333
24	24	1	1	0.5	1	333
25	25	1	1	0.0	5	303
26	26	1	1	0.1	5	303
27	27	1	1	0.2	5	303
28	28	1	1	0.3	5	303
29	29	1	1	0.4	5	303
30	30	1	1	0.5	5	303
31	31	1	1	0.0	5	313
32	32	1	1	0.1	5	313
33	33	1	1	0.2	5	313
34	34	1	1	0.3	5	313
35	35	1	1	0.4	5	313
36	36	1	1	0.5	5	313
37	37	1	1	0.0	5	323
38	38	1	1	0.1	5	323
39	39	1	1	0.2	5	323
40	40	1	1	0.3	5	323
41	41	1	1	0.4	5	323
42	42	1	1	0.5	5	323
43	43	1	1	0.0	5	333
44	44	1	1	0.1	5	333
45	45	1	1	0.2	5	333

StdOrder	RunOrder	Blocks	PtType	Concentration	Time	Temperature
46	46	1	1	0.3	5	333
47	47	1	1	0.4	5	333
48	48	1	1	0.5	5	333
49	49	1	1	0.0	10	303
50	50	1	1	0.1	10	303
51	51	1	1	0.2	10	303
52	52	1	1	0.3	10	303
53	53	1	1	0.4	10	303
54	54	1	1	0.5	10	303
55	55	1	1	0.0	10	313
56	56	1	1	0.1	10	313
57	57	1	1	0.2	10	313
58	58	1	1	0.3	10	313
59	59	1	1	0.4	10	313
60	60	1	1	0.5	10	313
61	61	1	1	0.0	10	323
62	62	1	1	0.1	10	323
63	63	1	1	0.2	10	323
64	64	1	1	0.3	10	323
65	65	1	1	0.4	10	323
66	66	1	1	0.5	10	323
67	67	1	1	0.0	10	333
68	68	1	1	0.1	10	333
69	69	1	1	0.2	10	333
70	70	1	1	0.3	10	333
71	71	1	1	0.4	10	333
72	72	1	1	0.5	10	333
73	73	1	1	0.0	24	303
74	74	1	1	0.1	24	303
75	75	1	1	0.2	24	303
76	76	1	1	0.3	24	303
77	77	1	1	0.4	24	303

StdOrder	RunOrder	Blocks	PtType	Concentration	Time	Temperature
78	78	1	1	0.5	24	303
79	79	1	1	0.0	24	313
80	80	1	1	0.1	24	313
81	81	1	1	0.2	24	313
82	82	1	1	0.3	24	313
83	83	1	1	0.4	24	313
84	84	1	1	0.5	24	313
85	85	1	1	0.0	24	323
86	86	1	1	0.1	24	323
87	87	1	1	0.2	24	323
88	88	1	1	0.3	24	323
89	89	1	1	0.4	24	323
90	90	1	1	0.5	24	323
91	91	1	1	0.0	24	333
92	92	1	1	0.1	24	333
93	93	1	1	0.2	24	333
94	94	1	1	0.3	24	333
95	95	1	1	0.4	24	333
96	96	1	1	0.5	24	333
97	97	1	1	0.0	48	303
98	98	1	1	0.1	48	303
99	99	1	1	0.2	48	303
100	100	1	1	0.3	48	303
101	101	1	1	0.4	48	303
102	102	1	1	0.5	48	303
103	103	1	1	0.0	48	313
104	104	1	1	0.1	48	313
105	105	1	1	0.2	48	313
106	106	1	1	0.3	48	313
107	107	1	1	0.4	48	313
108	108	1	1	0.5	48	313
109	109	1	1	0.0	48	323

StdOrder	RunOrder	Blocks	PtType	Concentration	Time	Temperature
110	110	1	1	0.1	48	323
111	111	1	1	0.2	48	323
112	112	1	1	0.3	48	323
113	113	1	1	0.4	48	323
114	114	1	1	0.5	48	323
115	115	1	1	0.0	48	333
116	116	1	1	0.1	48	333
117	117	1	1	0.2	48	333
118	118	1	1	0.3	48	333
119	119	1	1	0.4	48	333
120	120	1	1	0.5	48	333

Results and Discussion

Influence of concentration and temperature

Figures 2 shows the variation of inhibition efficiency with the inhibitor's concentration and solution's temperature at 5 hours immersion time. The figure clearly shows that the inhibitor's efficiency (inversely the corrosion rates) increases when added the inhibitor to the corrosive environment. This is thought to be due to an increase in the number of molecules that are adsorbed and spread over the mild steel surface.

This adds to the relative corrosion stability and resistance of the surface to corrosion. The inhibitor molecule used contains pairs of electrons on the sulfur, oxygen and nitrogen atoms in addition to resonance effect of the rings and inductive effects benzene ring and this enhances the inhibition efficiency of the inhibitor molecules.

On the other hand, the effect of temperature on the inhibition efficiency is as shown in the figure. Efficiency decreases with temperature. This is not helpful if we look at the corrosive environment under which mechanical parts work in Iraq where summer temperature rises above 50°C.

Generally, the inhibition efficiency of organic molecules depends on the adsorption/desorption behavior. Inhibiting activity of physisorbed inhibitors decreases with higher temperatures, but for chemisorbed inhibitors, the inhibition efficiency usually increases with higher temperatures [17, 18].

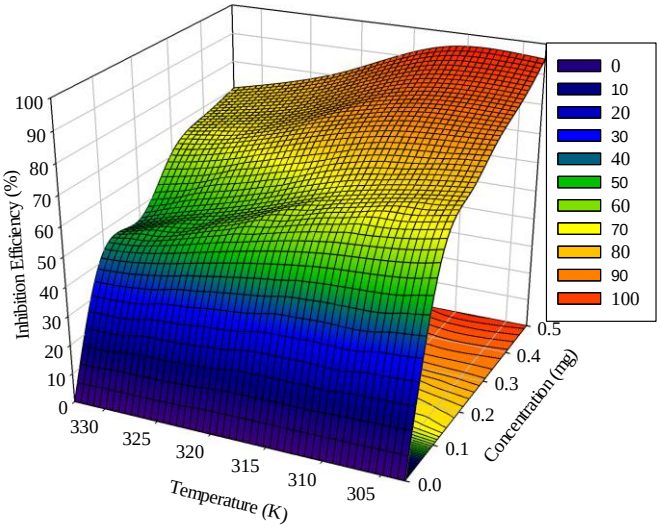

Figure 2. Effect of temperature on inhibition efficiency at different inhibitor concentration and 5 hr.

Influence of concentration and time

The influence of concentration and exposure time of the inhibition efficiency was studied. The obtained results are shown in Figure 3. In general, the corrosion rate (inferred from inhibition efficiency) value was found to decrease with. The higher temperature effect is to speed up the chemical reaction and to reduce oxygen solubility, which allows the occurrence of a cathodic reaction. The Figure also denotes the effect of the inhibitor with a reduction in the mild steel samples' corrosion rate value.

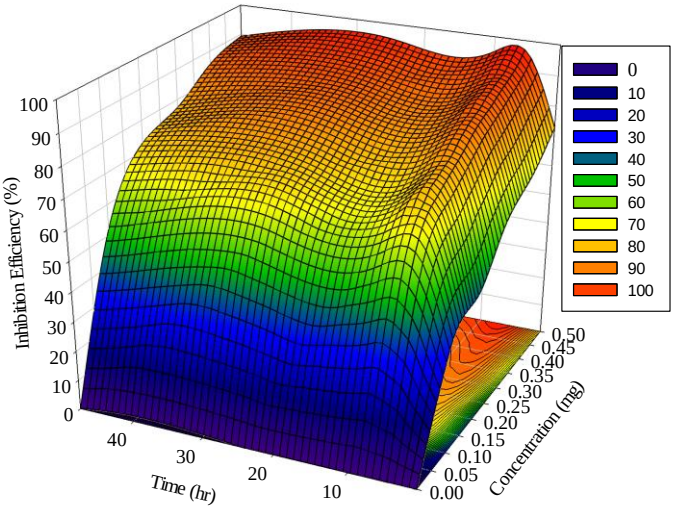

Figure 3. Effect of time on inhibition efficiency at different inhibitor concentrations and 303 K.

The temperature has a negative effect on the inhibitor efficiency, an increase in temperature results to increase of the dynamic energy for the inhibitor's molecules. The increase in dynamic energy slows the protective film formed on the surface of mild steel samples. With an increase in temperature, the corrosion rate and inhibitors' efficiency decrease.

The corrosion rate value was highest without inhibitor solution, and then it starts to decrease gradually with the presence of inhibitor as shown in Figure 3. However, inhibition efficiency increases with an increase of inhibitor concentration by a linear relationship.

The increase in IC leads to adsorption and surface coverage increase, therefore, leading to a reduction in corrosion rate values. The result indicates that the corrosion rate values decrease with the presence of inhibitor at all concentrations studied.

Influence of temperature and time

The influence of temperature and exposure time of the inhibition efficiency was studied. The obtained results are shown in Figure 4.

The results of the tests showed that the new corrosion inhibitor was effective in preventing the corrosion of mild steel in the acid environment up to 24 hours and still stable, as shown in Figure 4, but with increasing immersion time more than 24 hours the inhibition efficiency decreased [10, 19, 20].

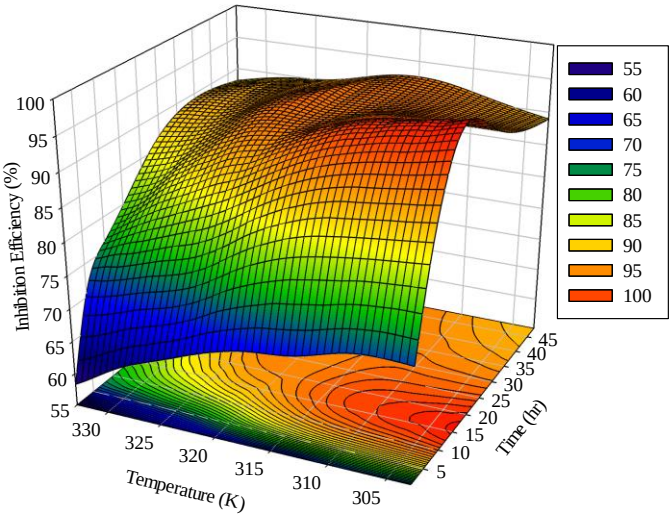

Figure 4. Effect of temperature on inhibition efficiency at different immersion time and 0.5 mg.

Development of regression models

Regression models were obtained for the response data which suggests a quadratic model. This regression model is a modified quadratic model from manual simplification and reduction of the model involving exclusion of high terms that are insignificant to obtain empirical model in terms of actual factors presented in Equation 6 as shown below:

$$Efficiency = -1548 + 935\ Concentration - 8.15\ Time + 9.80\ Temperature - 429.2\ Concentration \times Concentration - 0.03922\ Time \times Time - 0.01555\ Temperature \times Temperature - 0.215\ Concentration \times Time - 1.838\ Concentration \times Temperature + 0.03417\ Time \times Temperature \quad (6)$$

Both concentration and time are significant to first and second order, time is significant to the first order. Based on the interaction, all terms are significant except that for the interaction between concentration with time. A second-order effect of temperature is insignificant. These results are summarized below in Figure 5.

Table 3 also suggests that there is a strong interaction between the effects of concentration and temperature and insignificant interaction between concentration and time. However, the time and temperature seem to have significant interaction and effect on efficiency.

Table 3. Coded coefficients.

Term	Coef.	SE Coef.	95% CI	T-Value	P-Value	VIF
Constant	97.22	2.24	(92.78, 101.67)	43.34	0.000	
Concentration	32.68	1.36	(29.98, 35.38)	23.99	0.000	1.16
Time	17.48	1.19	(15.12, 19.84)	14.68	0.000	1.00
Temperature	4.25	1.25	(1.77, 6.72)	3.40	0.001	1.16
Concentration×Concentration	–26.83	2.16	(–31.11, –22.54)	–12.41	0.000	1.00
Time×Time	–21.66	2.26	(–26.13, –17.19)	–9.60	0.000	1.00
Temperature×Temperature	–3.50	1.94	(–7.35, 0.35)	–1.80	0.074	1.00
Concentration×Time	–1.26	1.74	(–4.71, 2.18)	–0.73	0.470	1.16
Concentration×Temperature	–6.89	1.69	(–10.25, –3.53)	–4.07	0.000	1.00
Time×Temperature	12.05	1.59	(8.89, 15.20)	7.56	0.000	1.16

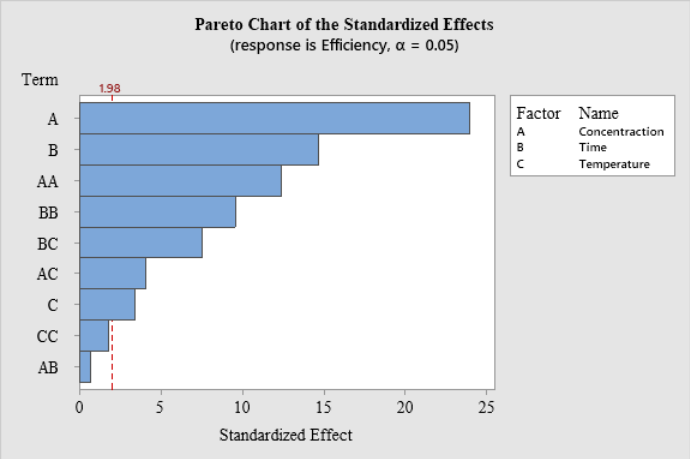

Figure 5. Pareto chart for the main effect.

Statistical analysis of mild steel corrosion using inhibitor

The accuracy and significance of the correlation are summarized in the following Table 4 and Figure 6.

Table 4. Model summary.

S	**R-sq**	**R-sq(adj)**
9.45035	96.65%	96.96%

The above table shows the accuracy of the model. The obtained result from this study shows that 96.96% of the inhibitor efficiency total variation relates to experimental variables. The selected factors represent the model obtained and describe the real relationship between the selected factors.

The P-value was used to check the model coefficient significance. The operation parameters were identified to be significant according to the model thereby influencing the responses. Hence, the corrosion of mild steel Type 316 in acidic solution has been proven statistically to depend on the operation parameters according to the frequency of each parameter occurrence in the model (Table 3).

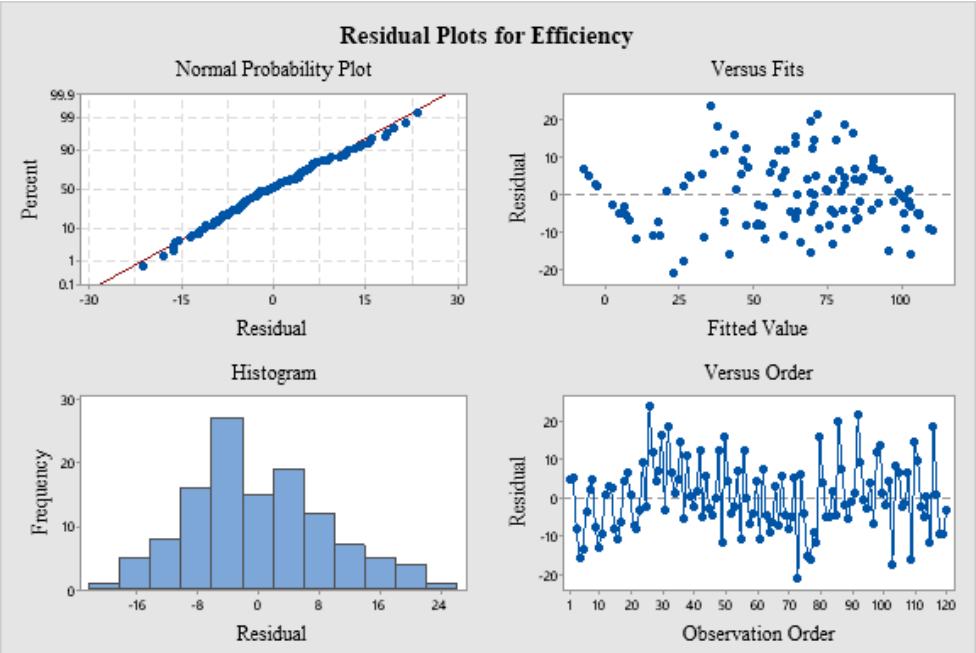

Figure 6. Residual plot for the inhibition efficiency.

Contour plots

Shown in Figure 7 contour 3-D response surface plot. In the 3-D response plots, the highest response value was ascribed to the factors in the design space as given with the clear peaks. These figures explain the surface behavior of variable combinations on mild steel

corrosion. The corrosion rate increases when the temperature increases and the inhibitor concentration decreases.

Figure 7-A shows that temperature and inhibitor efficiency were significant on the response, but concentration has a more significant influence than solution temperature. The interaction between both factors was more significant with a negative effect on the response which indicates that an increase in the level of both variables is an inefficient method to reduce *CR* (also shown in as shown in Figure 2). It also shows that inhibition efficiency increase when the temperature decreases and the inhibitor concentration increase beyond 0.3 g.

At low exposure time, the inhibition efficiency (shown in Figure 7-B) value increased with inhibitor concentration. However, a significant increase in the inhibition efficiency was noticed when inhibitor concentration increased beyond 0.3 g and time was increased beyond 20 hrs.

Figure 7-C suggests that at low concentrations, temperature becomes effective at higher levels of exposure time is increased beyond 25 hrs.

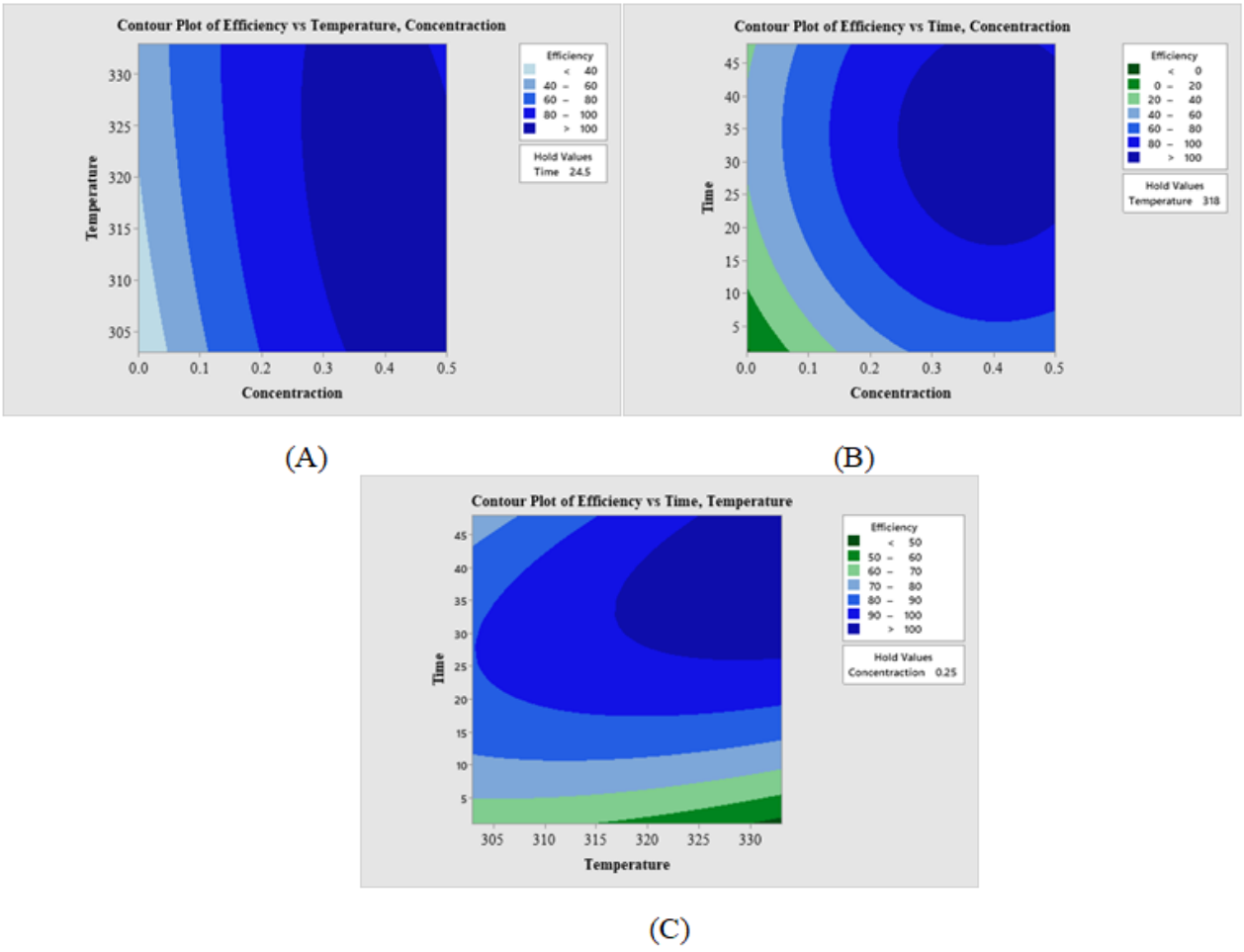

Figure 7. Contour surface plot for the variables.

Optimization using desirability approach

The last main aim of this study was to find the optimum parameters of the process to minimize the corrosion rate from the developed mathematical model equations.

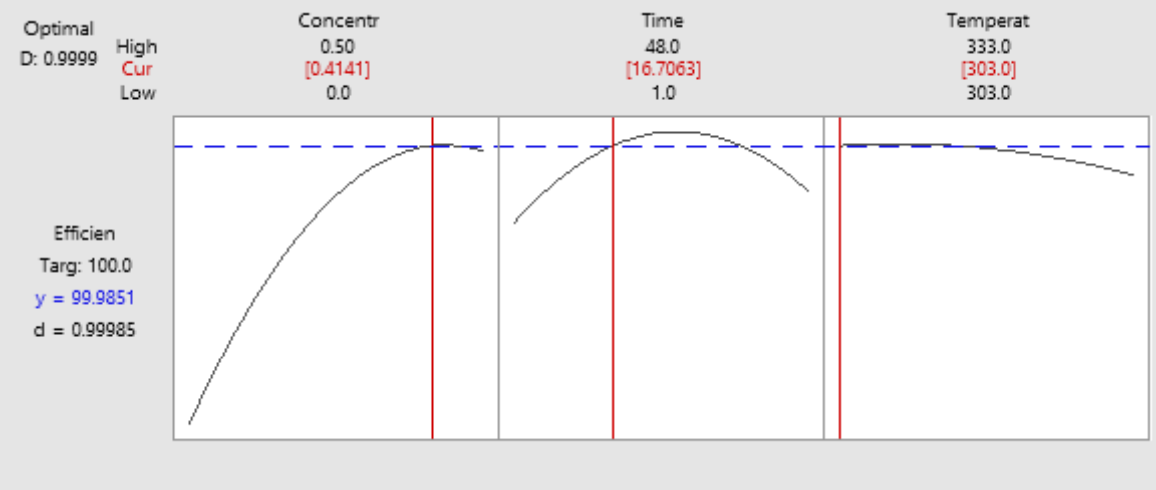

Figure 8. Optimization of variables.

Response surface method via desirability optimization approach was applied. The optimization of the variables was done withing the range studied that gives minimum corrosion rate. The optimum conditions for getting the highest inhibition efficiency (lowest corrosion rate) at static conditions is when the temperature value is equal to 30°C, exposure time 16 h and the inhibitor concentration is 0.4 g/L, as shown in Figure 8.

Conclusion

Conclusions drawn from the experimental and theoretical results are as follows:

1. An increase in temperature leads to increased corrosion rate and reduced inhibition efficiency.
2. The response surface methodology quadratic model developed showed that temperature and exposure time significantly affects the inhibitor efficiency in sodium chloride solution.
3. The optimum process variable from the quadratic model developed was 0.4141 g inhibitor concentration, 16.7063 h exposure time, and temperature of 30°C with a predicted optimum inhibitor efficiency value of 100% using the surface response method for the experiment design.
4. The obtained results from the optimum value validated agree with predicted values by the quadratic model.

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