Utilization of Experimental Design as an Approach to Optimize Inhibition Efficiency of a Pyridazine Derivative Against Mild Steel Corrosion in 1 M Hydrochloric Acid

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Abstract
The aim of this manuscript was to optimize the IE(%) of a pyridazine derivative (MDP) for hindering MS corrosion in a 1 M HCl solution. To identify the most influential parameters affecting CI, a study was conducted using a Doehlert matrix and Nemrod software. Based on preliminary knowledge, three parameters (Ct from MDP, IT and T) were selected. IE(%) was evaluated by analyzing PDP intensity-potential curves and interpreting the obtained results. The novelty of this research stems from the methodical application of DoE methodology to optimize CI, which offers notable advantages such as reduced test requirements, and the ability to discern interactions between factors. The findings revealed a remarkable IE(%) of 96%, under optimal conditions: Ct from MDP of 0.3×10^{-3} mol/L, IT of 12 h and T of 303.15 K.

Keywords: corrosion; DoE; inhibition; MDP; MS; PDP.

Introduction•

DoE plays a crucial role in industrial research and development studies across various fields, such as petrochemical, pharmaceutical, metallurgical and chemical industries [1-7]. This method is a general approach to improve quality, reduce the number of trials, detect interactions between factors, model the studied response and achieve optimal precision of results. As environmental concerns continue to grow, steel's ecological properties are becoming increasingly valued, since its magnetic properties enable it to be recovered from waste and separated from other materials. Thus, it is a highly recyclable material [8-10].

•The abbreviations list is in page 163.

Recycling steel does not change its properties, and contributes to significantly reduce the amount of household waste, while preserving the natural resources of iron ore [11-13]. In today's world, where the environment is highly valued, steel's ecological properties are much appreciated.
Steel also offers many advantages for the construction industry [14]. It is an extremely hard material, yet flexible, and it can undergo significant deformation before breaking. It can withstand heavy weights and it is shock-resistant. When treated by galvanization, steel becomes an anti-corrosive material that requires little maintenance, and is non-combustible, reducing fire hazards. Its resistance to earthquakes is also notable.
CI are among the most commonly used methods to prevent steel oxidation in an acidic medium, particularly in pickling baths. Organic compounds rich in rings and heteroatoms are excellent CI [15-19].
Herein, a Doehlert matrix was employed for systematically organizing experimental trials to optimize processes, identify the most influential factors and minimize the number of necessary trials. Nemrod software was used for the variance calculation and contour plot creation.

Materials and methods

CI

In this study, a pyridazine derivative known as MDP (Fig. 1) was employed as CI.

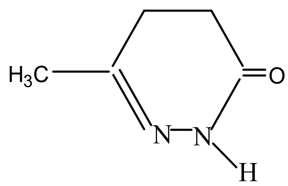
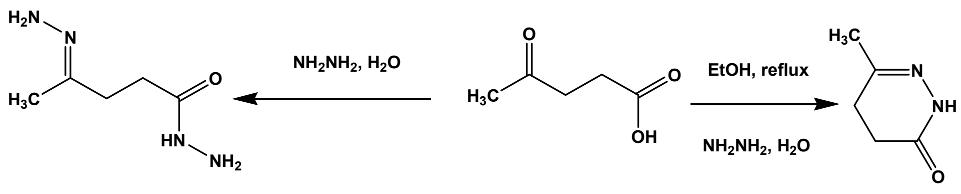


Figure 1: Molecular structure of MDP.

MDP was achieved through the following synthesis route (Scheme 1):



Scheme 1: MDP synthesis route.

Electrolytes

A HCl solution, graded at 37% Ct, with a density of 1.19, from Riedel Haen, was diluted with distilled water, to prepare 1 M of the corrosive solution. The normality was regulated through acid-base titration.

Specimens

The chemical composition of the MS herein used is presented in Table 1.

Table 1: Chemical composition of MS.

Element	Fe	C	Si	M	Cr	Mo	Ni	Al	Cu	Co	V	W
%	98.7	0.11	0.24	0.47	0.12	0.02	0.1	0.03	0.14	<0.0012	<0.003	0.06

MS samples, measuring 1 × 5 × 0.06 cm, were physically polished using an abrasive paper to ensure a uniform surface before each experiment. Acetone was employed to degrease the samples and remove any contaminants.

I-E polarization curves

PDP curves were drawn using a PGZ 100 potentiostat, employing an electrochemical cell with a MS sample (1 cm^2), a Pt plate and saturated calomel as working, auxiliary and reference electrodes, respectively.
PDP studies were conducted with a scanning speed of 1 mV/s^{-1}, within the potential range from -750 to -100 mV, relative to E_{corr} [20-22]. IE(%) from MDP is defined by eq. (1) [23, 24]:

$$IE\% = \frac{I_{corr} - I_{corr}^{inh}}{I_{corr}} \times 100 \qquad (1)$$

where I_{corr} and I_{corr}^{inh} represent values determined by Tafel straight lines extrapolation in a 1 M HCl medium, without and with MDP, respectively.

Results and discussion

Model used

Using a second-degree model, four variables were herein examined, and ten coefficients were calculated. To estimate Y response variable in this model, a second-degree polynomial was employed [25, 26].

$$Y = b_0 + b_1 X_1 + b_2 X_2 + b_3 X_3 + b_{11} X_1^2 + b_{22} X_2^2 + b_{33} X_3^2 + b_{12} X_1X_2 + b_{13} X_1X_3 + b_{23} X_2X_3 \qquad (2)$$

Terms belonging to 4 families were estimated: constant term (b_0); first degree term (bi); square term (bii); and rectangle term (bij).
To calculate the model's coefficients, the following matrix system had to be solved:

$$Y = X B \qquad (3)$$

where Y, X and B are the responses, the model's and the coefficients matrixes, respectively. The resolution of this system by the method of least squares was obtained by the following formula:

$$B = ({}^tX X)^{-1}\, {}^tX Y \qquad (4)$$

where ${}^tX$ is the transposed matrix of X.

Uniform Doehlert networks

Estimation of the coefficients for the quadratic model was performed using an experimental plan based on uniform Doehlert networks. This matrix, generated from a simplex, provided a uniform distribution of tests across the entire experimental domain, and allowed assigning different levels to independent variables, based on their importance (Table 2).

Table 2: Matrix of experiments.

Experiment number	X_1	X_2	X_3
1	1.0000	0.0000	0.0000
2	-1.0000	0.0000	0.0000
3	0.5000	0.8660	0.0000
4	-0.5000	-0.8660	0.0000
5	0.5000	-0.8660	0.0000
6	-0.5000	0.8660	0.0000
7	0.5000	0.2887	0.8165
8	-0.5000	-0.2887	-0.8165
9	0.5000	-0.2887	-0.8165
10	0.0000	0.5774	-0.8165
11	-0.5000	0.2887	0.8165
12	0.0000	-0.5774	0.8165
13	0.0000	0.0000	0.0000
14	0.0000	0.0000	0.0000
15	0.0000	0.0000	0.0000
16	0.0000	0.0000	0.0000

The results of this study were obtained by 16 experiments conducted with 4 tests repetitions in the domain centre, to account for experimental errors [27, 28]. DoE were allowed to predict the responses of each experiment, which could be visualized in the form of contour plots using Nemrod software [6].

By setting a parameter, typically at the domain centre, the evolution of each response can be tracked. The use of contour plot allows for easy data interpretation, as they provide a clear visualization of the relationship between experimental variables and responses [29]. By analysing the curves, researchers can determine which variables have the greatest impact on the responses, and identify optimal conditions for achieving desired outcomes. Additionally, the inclusion of four repetitions in the domain centre helps to reduce experimental errors and improves the accuracy of results.

Experimental domain

The results of the study showed that CI from MS in HCl can indeed be influenced by the Ct of CI, T and IT of samples. The experiments were conducted by varying these three factors and observing resulting IE(%). Findings indicated that higher Ct of CI generally led to higher IE(%), while increasing T had a negative effect on it [30, 31]. IT effect on IE(%) was found to be less significant than that from other two factors, but still had an impact on it [30].

These findings are important, since they provide insight into the optimal conditions for CI of MS in an acidic medium, which can have practical implications in industries that use acidic solutions. The results suggest that careful control of the Ct of CI and T is critical for achieving high IE(%), while IT can be adjusted within a reasonable range, without a significant impact. By understanding the influence of these factors, researchers and practitioners can design more effective inhibition strategies and optimize the use of CIs in acid solutions. The findings allowed to establish the experimental domain (Table 3).

Table 3: Experimental domain of interest.

	Factor	Unit	Center	Step variation
U_1	Ct from CI	mol/L (x 10^{-3})	0.155	0.145
U_2	T	K	318.15	288.15
U_3	IT	h	6.25	5.75

Responses studied

The response variable for the CI is IE(%), which was obtained from I-E curves. This was the aim of another study by [7], who have researched MDP's mechanism and mode of action for corrosion resistance [7].

<u>IE(%) study</u>

The experimental plan was obtained by directly applying the model herein used. Measured responses for IE(%) (Y) are presented in Table 4.

Table 4: Experimentation plan corresponding to Doehlert matrix.

Experiment no.	Ct (mol/L x10^{-3})	T (K)	IT (h)	IE(%)
1	0.3000	318.1500	6.2500	91
2	0.0100	318.1500	6.2500	74
3	0.2275	331.1400	6.2500	90
4	0.0825	305.1600	6.2500	87
5	0.2275	305.1600	6.2500	94
6	0.0825	331.1400	6.2500	70
7	0.2275	322.4805	10.9449	87
8	0.0825	313.8195	1.5551	82
9	0.2275	313.8195	1.5551	85
10	0.1550	326.8110	1.5551	83
11	0.0825	322.4805	10.9449	81
12	0.1550	309.4890	10.9449	89
13	0.1550	318.1500	6.2500	83
14	0.1550	318.1500	6.2500	82
15	0.1550	318.1500	6.2500	85
16	0.1550	318.1500	6.2500	84

<u>Calculation of coefficients using coded variables</u>

Coefficients calculation used coded variables with Nemrod software, of which results are shown in Table 5. It is worth noting that the number of conducted tests was significantly greater than the number of coefficients to be calculated. Specifically, the model contained 10 coefficients, while the experimental plan involved 16 tests. This means that the number of DoF was equal to the difference between the number of tests and coefficients, which was 16 - 10 = 6. This information about the DoF is important, as it helps to determine the statistical significance of the results. In this case, with six DoF, the statistical analysis could be performed using an appropriate distribution (such as the t-distribution), to determine whether the coefficients were significantly different from zero.

Table 5: Estimation of the coefficients of the postulated quadratic model.

Name	Coefficient	F. inflation	Ecart-type	T. exp.	Signif. %
b_0	83.500	-	0.645	129.36	***
b_1	8.750	1.00	0.645	13.56	***
b_2	-5.340	1.00	0.645	-8.27	**
b_3	1.429	1.00	0.645	2.21	11.3%
b_{11}	-1.000	1.13	1.118	-0.89	43.9%
b_{22}	2.667	1.13	1.118	2.39	9.6%
b_{33}	1.083	1.11	1.054	1.03	38.1%
b_{12}	7.506	1.11	1.491	5.03	*
b_{13}	-0.817	1.11	1.667	-0.49	65.7%
b_{23}	-1.885	1.11	1.667	-1.13	34.1%

The applied model is a multiple linear regression model, of which equation is as follows:

$$Y = 83.5 + 8.75\, X_1 - 5.34X_2 + 1.43X_3 - X_1^2 + 2.67X_2^2 + 1.03X_3^2 + 7.51X_1X_2 - 0.82X_1X_3 - 1.88X_2X_3 \quad (5)$$

In this model, Y represents the dependent variable (responses for IE(%)); X_1, X_2 and X_3 are independent variables; and X_1^2, X_2^2 and X_3^2 represent the squared terms of the respective independent variables. The other terms in the model are interaction terms among independent variables. The coefficients associated with each variable and term in the model represent their impact on the dependent Y variable.

Sd of the response is a measure of experimental errors, and it indicates the uncertainty of each test. Directly estimating Sd allows for a better understanding of data precision and results reliability.

Sd of each coefficient can also be calculated to determine whether it is statistically different from 0. This is performed by dividing the coefficient by its Sd, and comparing the result to the values of a Student's t distribution. The fourth column in the output shows the coefficient Sd, and the next column shows t criterion. The software used in the analysis provided the probability associated with the Student's t value for a given number of DoF, which represents the risk α of being wrong by rejecting the hypothesis that the coefficient is zero.

Herein, it was decided to consider a coefficient significant if the associated probability was less than or equal to 0.05 or 5% [32, 33]. This means that a 5% risk of being wrong was accepted, by rejecting the hypothesis that the coefficient was zero. The significant coefficients in this study were b_1, b_2 and b_{12}, which have associated probabilities greater than or equal to 0.05.

Doehlert experimental design has been used by [34] to assess 3-((dicyclohexylamino)methyl)-5-(4-((3,4-dimethoxybenzylidene)amino)phenyl)-1,3,4-oxadiazole-2(3H)-thione efficiency as CI against MS corrosion in a 1 M HCl solution. Electrochemical measurements revealed an enhancement in IE(%) with higher Ct from CI. Specifically, combined influences of Ct from CI and T exhibited the most significant effect [34].
In another study employing a similar approach to optimize the effectiveness of a CI made of 1-propanol against MS corrosion in a 1 M acidic environment, Doehlert experimental design was also utilized. It was seen that both the Ct of the CI and T had a very significant effect on the inhibitor effectiveness [35].
Thus, statistical analysis of experimental data involves calculating Sd values for the responses and each coefficient, to determine the precision and reliability of the results. The significance of each coefficient was determined by comparing its value to the values of a Student's t distribution, and calculating the associated probability. The initial model was ultimately simplified to a model such as:

$$Y = 83.5 + 8.75X_1 - 5.34X_2 + 1.43X_3 + 7.51X_1X_2 \quad (6)$$

Regression variance analysis was used to interpret the results of a calculation program. The effect of the regression model was compared to the residual effect, and it was used to assess the model significance.
The calculation principle involves decomposing SCE of the differences into two components: SCE due to the model and residual SCE [36, 37]. The variances corresponding to these two sources of variation were then calculated and compared by a Fischer test. If the variance due to the regression is greater than the residual variance, it can be concluded that the model is significant [38].
In other words, regression variance analysis provides a way to assess the overall fit of the regression model by comparing the variation explained by the model with the variation not explained by the model (residual variation). If the variance explained by the model is significantly greater than the residual variance, it indicates that the model is significant and it has a good fit to the data. On the other hand, if the residual variance is significantly greater than the variance explained by the model, it suggests that the model is not a good fit, and may need to be revised or discarded.
Overall, regression variance analysis provides a useful tool for interpreting the results of a regression analysis and assessing the model significance. By comparing the variance due to the regression with the residual variance, one can determine the overall fit of the model, and make informed decisions about its use and application.

<u>Analysis of variance</u>
Table 6 gives the results of this analysis for the obtained measurements. The value of Fischer ratio corresponds to variables DoF 9 and 10. The probability associated with this value is less than 0.001.

Therefore, it can be concluded that the chosen model provides a very statistically significant explanation for the variations in the risk response of 5%.

Table 6: Variance analysis table.

Source of variation	SCE	Degrees of liberty	Medium square	Report	Signif
Regression	492.4348	9	54.7150	32.8290	**
Residues	52.0027	6	8.6671		
Validity	47.0027	3	15.6676	9.4005	*
Error	5.0000	3	1.6667		
Total	544.4375	15			

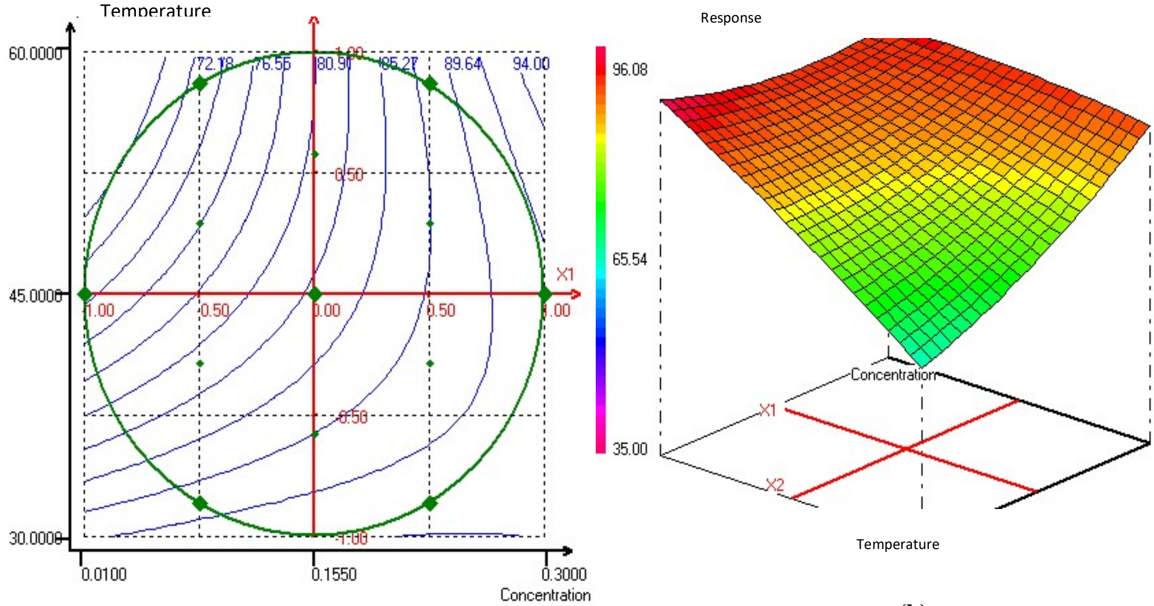
One important aspect of the analysis is using the coefficient of determination to evaluate the performance of the chosen second-degree model. This measure gave the percentage of the total variance in the dependent variable Y, which is explained by the model. In this case, the coefficient of determination was found to be 0.92, indicating that the chosen model explained 92% of the variance in Y, which is considered to be a satisfactory result [40, 41].
The estimation of the coefficients for the different models is shown in Table 5.
However, it is not enough to know the estimated coefficients for determine if they have a significant influence on the phenomenon under observation. To achieve this, the analysis used a Student t test to determine if the estimated coefficients, denoted as bi, are significantly different from zero [39]. If a coefficient is significantly different from zero, it has a significant influence on the phenomenon being observed, and if not, it can be considered insignificant.

Optimal research and interpretation

The theoretical model finally preserved is:

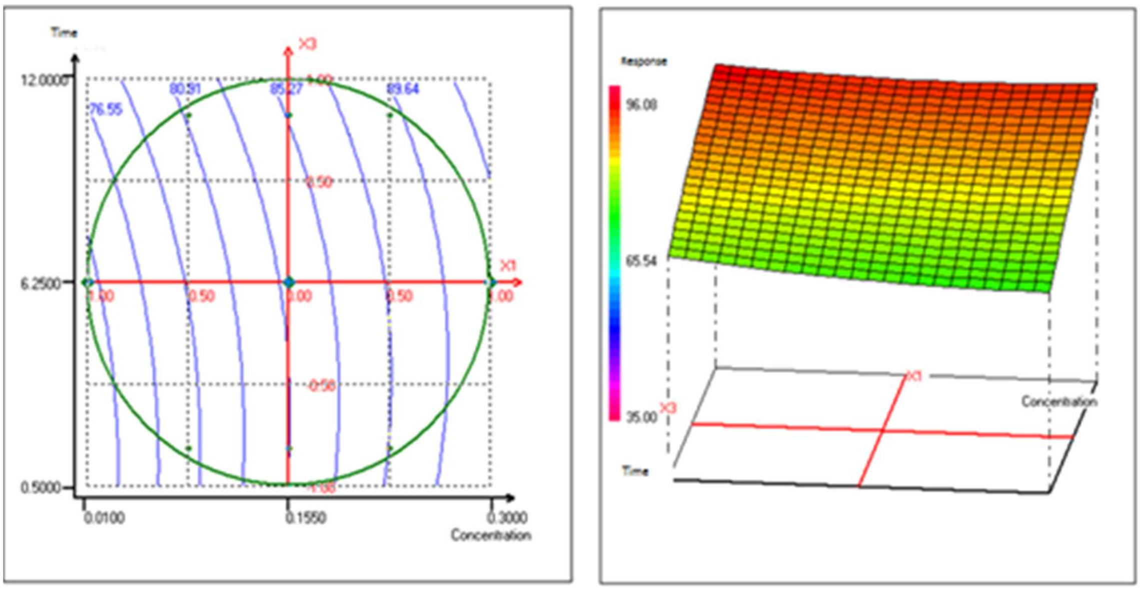
$$Y = 83.5 + 8.75X_1 - 5.34X_2 + 1.43X_3 + 7.51X_1X_2 \quad (7)$$

The model applied was validated, and the expected response can be calculated at any point within the experimental area of interest. This information can be visualized by plotting the calculated response values as points on a graph, and connecting them to form contour plots. These curves can be easily represented in a two- or three-dimensional space [42].
However, the analysis is limited to those coefficients that have an effect on IE(%): Ct, IT and T (Figs. 2-4). This variance may be seen in the contour plots, which show IE(%) as a function of Ct from CI and IT. Furthermore, the observed decrease in IE(%) with higher T suggests that the latter may have a negative effect on the former. This information could be useful for developing strategies to optimize the use of CI, e.g., by operating at lower T to achieve higher IE(%). Overall, the results highlight the importance of carefully controlling both the Ct from CI and T to achieve optimal IE(%).



(a) **(b)**

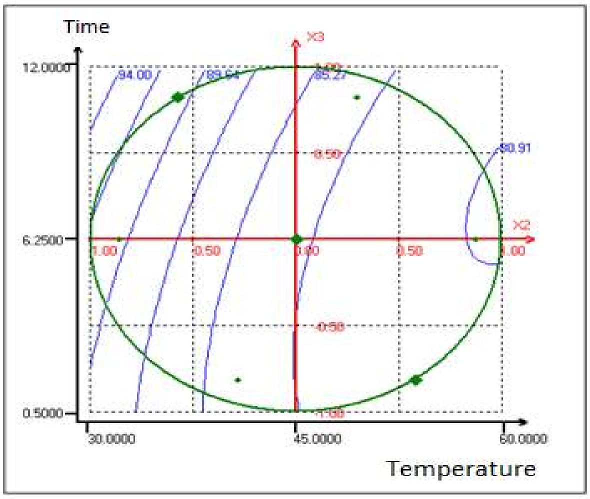
Figure 2: Efficiency of the plan for **(a)** variables (Ct and T) and **(b)** fixed factor (IT of 6.25 h).



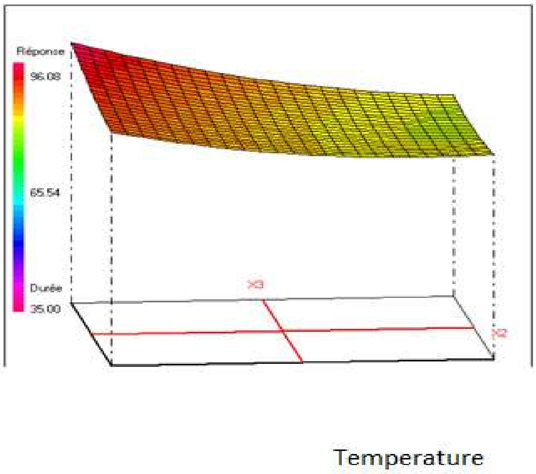
(a) **(b)**

Figure 3: Efficiency of the plan for **(a)** variables (Ct and IT) and **(b)** fixed factor (T of 318.15 K).

This information can be useful for developing strategies to optimize the use of the CI. For example, if one desires to achieve a high IE(%), it may be beneficial to increase the IT, while keeping the Ct from CI and T constant. This can lead to a higher IE(%) and better control over the system being inhibited.



(a)



(b)

Figure 4: Efficiency of the plan for **(a)** variables (T and IT) and **(b)** fixed factor (Ct of 0.1550×10^{-3} mol/L).

Overall, the results suggest that carefully controlling the IT be an important factor for achieving optimal IE(%). They also highlight the need to consider the effects of other variables, such as Ct from CI and T, for developing effective inhibition strategies.

The results show that there was a variation in IE(%), when T and IT were changed, at a constant Ct from CI of 0.1550×10^{-3} mol/L. It was seen that IE(%) decreased with higher T, indicating that the latter negatively affected IE(%). On the other hand, IE(%) increased with higher IT, which shows a positive correlation between the two. These findings can be useful for developing optimal CI. For example, if high IE(%) is desired, it may be beneficial to increase IT while keeping T constant. However, if T cannot be controlled, a higher Ct of CI may be required to maintain a desired level of IE(%).

Overall, the results highlight the importance of carefully controlling both T and IT to achieve optimal IE(%) when using a fixed Ct of the CI. These findings can inform the development of effective strategies for the use of CI in various industrial applications.

Conclusion

The conducted experiment demonstrated the exceptional effectiveness of MDP as CI in a 1 M HCl solution. The experiment's methodology was meticulously developed to minimize the number of required tests, thereby saving both time and resources. The mathematical model derived from the experiment showed a high level of accuracy, enabling reliable forecasts of corrosion rates within the experimental domain. Analysis of the obtained data led to the identification of optimal inhibition conditions, characterized by an IT of 12 h, Ct from MDP of 0.3×10^{-3} mol/L and T of 303.15 K.

Through the employed methodology, the best operating conditions for achieving a maximum IE(%) of 96% were determined. Consequently, MDP demonstrates immense potential for practical applications in corrosion prevention within acidic environments.

Authors' contributions

Forsal Issam: conceived and designed the analysis; collected the data; inserted data; wrote the original draft; project administration. **Mernari Bouchaib**: contributed to conceptual; performed the analysis; contributed to the interpretation of the results. **Lahmady Sara:** literature review and editing; collected the data. **Elkhotfi Yassine**: conceived and designed the analysis; edited and formatted the paper. **Benbouya Khalid**: contributed to the interpretation of the results; resources and validation.

Abbreviations

CI: corrosion inhibitor
Ct: concentration
DoE: design of experiments
DoF: degrees of freedom
$\mathbf{E_{corr}}$: corrosion potential
HCl: hydrochloric acid
$\mathbf{I_{corr}}$: corrosion current density
IE(%): inhibition efficiency
IT: immersion time
MDP: 6-methyl-4,5-dihydropyridazin-3(2H)
MS: mild steel
PDP: potentiodynamic polarization
SCE: sum of the squares
Sd: standard deviation
T: temperature

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