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Use of the experimental designs as an approach to optimize the inhibition efficiency of a Pyridazine derivative against corrosion of steel in an acidic medium

Issam Forsal, Mernari Bouchaib[1], Elkhotfi Yassine[1], Benbouya Khalid[2]

1 University Sultan Moulay Sliman
2 Mohammed V University

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Abstract

In this manuscript, our aim was to optimize the inhibitory performance of a pyridazine derivative against steel corrosion in a 1M HCl solution. The percentage of inhibitor efficiency depends on various parameters, including inhibitor concentration, solution temperature, and immersion time. Using the Dohlert matrix and NemrodW software, we conducted a study to determine the most influential parameters affecting the corrosion phenomenon. Based on preliminary knowledge, we selected three parameters: inhibitor quantity, immersion time, and temperature. We evaluated the inhibitory efficiency by analyzing the Potentialdynamic intensity-potential curves and discussing the results obtained. Experimental designs offer benefits such as reducing the number of tests and detecting interactions between factors. By following the experimental design methodology, we aimed to determine the best conditions to obtain maximum inhibitor efficiency while reducing the number of required tests.

Forsal Issam[1*], **Mernari Bouchaib**[1], **Elkhotfi Yassine**[1], **Benbouya Khalid**[2]

[1] *Laboratory of Engineering and Applied Technologies, Height School of Technology, Sultan Moulay Slimane University Beni Mellal Morocco.*

[2] *EMDD_CERNE2D, Height School of Technology, Mohammed V University in Rabat, Sale Morocco.*

[*]E-mail: forsalissam@yahoo.fr

Abstract

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1. Introduction

Experimental designs play a crucial role in industrial research and development studies across various fields such as petrochemical, pharmaceutical, metallurgical, and chemical industries [1][2][3][4][5][6][7]. The use of experimental plans is a general approach aimed at improving quality with the following objectives: reducing the number of trials, detecting interactions between factors, modeling the studied response, and achieving optimal precision of results.

As environmental concerns continue to grow, the ecological properties of steel are becoming increasingly valued. Steel's magnetic properties enable it to be recovered from waste and separated from other materials, making it a highly recyclable material [8][9][10]. The recycling of steel has no effect on its properties, which remain unchanged, and this can significantly reduce the amount of household waste while preserving the natural resources of iron ore [11][12].

In today's world where the environment is highly valued, steel's ecological properties are appreciated. Steel, with its magnetic properties [13], can be recovered and separated from all waste, making it infinitely recyclable and preserving natural resources of iron ore while reducing household waste [8][9][10]. Steel also offers many advantages in the construction industry [14]. It is an extremely hard material, yet flexible and can undergo significant deformation before breaking. It can withstand heavy weights and is shock-resistant. When treated by galvanizing, steel becomes an anticorrosive material that requires little maintenance and is non-combustible, reducing the risk of fire. Steel's resistance to earthquakes is also notable. Inhibitors are among the most commonly used methods to avoid oxidation of steel in an acid medium, particularly in pickling baths. Organic compounds rich in rings and heteroatoms have excellent corrosion inhibitors [15][16][17][18][19][20][21][22][23][24][25][26][27][28][29][30][31][32][33][34][35][36][37][38][39].

In this study, we optimized the efficiency inhibition of a pyridazine derivative against the corrosion of ordinary steel in a 1M hydrochloric acid medium using a Dohlert matrix. The calculation of variance and drawing of iso-response diagrams were done using the NemrodW software.

2. Materials and methods

2.1. Inhibitor

The inhibitor is a Pyridazine derivative: 6-methyl-4,5-dihydropyridazin-3(2H) one named (CDM).

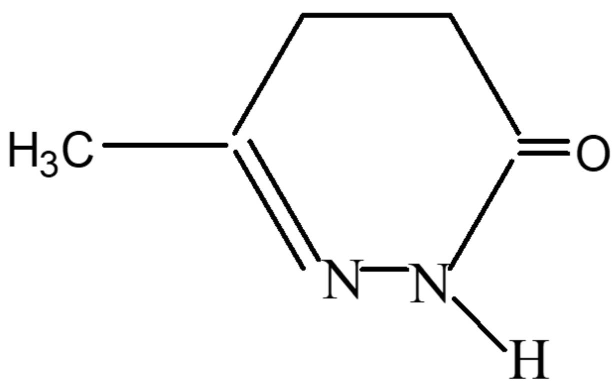
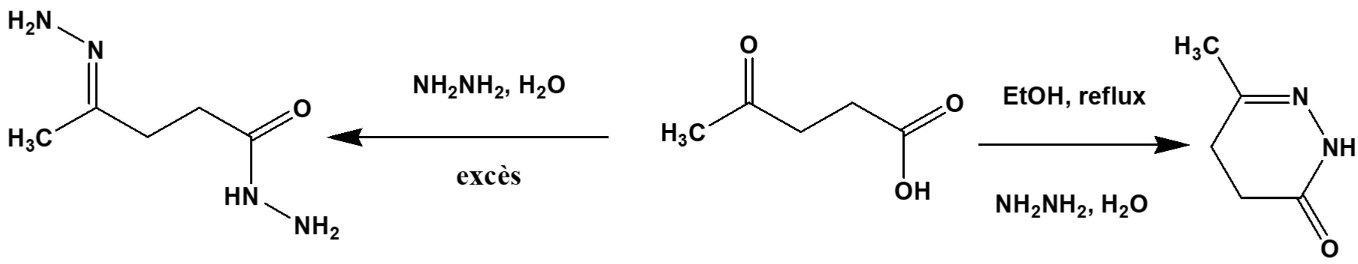


Figure 1. Molecular structure of 6-methyl-4.5 dihydropyridazin-3(2H)-one (CDM)

The (CDM) is achieved through the following mechanism:



2.2. Electrolytes

The solutions HCl 1M have been prepared from the dilution of a solution of marketed HCl brand Riedel Haen with density d = 1.19 and percentage 37%. Normality is controlled by acido-basic dosage.

2.3. Specimens

The following table presents the chemical composition of the ordinary steel used in this study:

Element	Fe	C	Si	Mn	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

Table 1. The chemical composition of ordinary steel.

To prepare for each experiment, the steel samples, which have dimensions 1 cm^2, are mechanically polished using abrasive paper to ensure a homogeneous surface. Additionally, the samples are degreased with acetone to remove any impurities.

2.4. I-E polarization curves

To draw the Potentialdynamic curves, we used a Potensiostat PGZ 100. The electrochemical cell contained three electrodes, with the 1cm surface steel serving as the working electrode, a platinum plate serving as the auxiliary electrode, and a saturated calomel electrode serving as the reference electrode. Potentialdynamic polarization studies were carried out with a scanning speed of 1 mV/s in the potential range of -750 mV to -100 mV, relative to the corrosion potential [40].

The inhibition efficiency of the compound is defined by the relationship:

$$E\% = \frac{I_{corr} - I_{corr}^{inh}}{I_{corr}} \times 100\%$$

Where I_{corr} and I_{corr}^{inh} represent, the corrosion current densities determined by the extrapolation of Tafel straight lines in 1M HCl medium, respectively, with and without inhibitor.

3. Results and discussion

3.1. Model used

In our study, we investigated four factors, and to estimate 15 coefficients, we used a second-degree model, which involves estimating the Y response using a second-degree polynomial.

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

You will have to estimate terms belonging to 4 families:

- b_0: constant term
- bi: first degree term
- bii: square term
- bij: rectangle term

To calculate the coefficients of the model, we have to solve the following matrix system:

Y = X B

With:

- Y: matrix of responses
- X: matrix of the model
- B: matrix of coefficients

The resolution of this system by the method of least squares is obtained by the following formula:

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

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

3.2. Uniform Doehlert Networks

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

Table 2. Matrix of experiences

Experience 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 indicate that 16 experiments were conducted, with 4 repetitions of the test in the center of the domain to account for experimental error. The responses of each experiment were predicted using the experimental design and can be visualized in the form of iso-response curves using the NemrodW software {m/69/}. By setting a parameter, typically at the center of the domain, the evolution of each response can be tracked.

The use of iso-response curves allows for easy interpretation of the data, providing a clear visualization of the relationship between the experimental variables and the response. By analyzing the curves, researchers can determine which variables have the greatest impact on the response and identify the optimal conditions for achieving the desired response. Additionally, the inclusion of four repetitions in the center of the domain helps to reduce experimental error and improve the accuracy of the results.

3.3. Experimental Domain

The results of the study showed that the inhibition of steel in an acidic medium can be influenced by the amount of inhibitor, temperature, and immersion time of samples. The researchers conducted experiments by varying these three factors and observing the resulting inhibition efficiency. The results indicated that increasing the amount of inhibitor generally led to higher inhibition efficiency, while increasing the temperature had a negative effect on inhibition efficiency. The effect of immersion time was found to be less significant than the other two factors but still had an impact on inhibition efficiency.

These findings are significant because they provide insight into the optimal conditions for inhibiting steel in an acidic medium, which can have practical implications in industries that use acid solutions. The results suggest that careful control of the amount of inhibitor and temperature is crucial for achieving high inhibition efficiency, while the immersion time can be adjusted within a reasonable range without a significant impact on efficiency. By understanding the influence of these factors, researchers and practitioners can design more effective inhibition strategies and optimize the use of inhibitors in acid solutions. Based on our preliminary results, we were able to define the experimental domain (see Table 3).

Table 3. The experimental domain of interest

	Factor	Unit	Center	step variation
U_1	Inhibitor concentration	mmol/l	0.155	0.145
U_2	Temperature	°C	45	15
U_3	Time	h	6.25	5.75

- *Responses studied*

The response variable for the inhibitor is the inhibition efficiency (E%), which is obtained from the I-E curves. This was the aim of another study [41]. Additionally, the mechanism and mode of action of this compound in the behavior of corrosion resistance were also studied in the aforementioned article.

- *Inhibition efficiency study*

The experimental plan was obtained by directly applying the model used in our study. The measured responses for inhibition efficiency (Y) are presented in Table 4.

Table 4. Experimentation plan corresponding to Doehlert matrix

Experience number	Concentration	Temperature	Time	Efficiency
	mmol	°C	h	%
1	0.3000	45.0000	6.2500	91.00
2	0.0100	45.0000	6.2500	74.00
3	0.2275	57.9900	6.2500	90.00
4	0.0825	32.0100	6.2500	87.00
5	0.2275	32.0100	6.2500	94.00
6	0.0825	57.9900	6.2500	70.00
7	0.2275	49.3305	10.9449	87.00
8	0.0825	40.6695	1.5551	82.00
9	0.2275	40.6695	1.5551	85.00
10	0.1550	53.6610	1.5551	83.00
11	0.0825	49.3305	10.9449	81.00
12	0.1550	36.3390	10.9449	89.00
13	0.1550	45.0000	6.2500	83.00
14	0.1550	45.0000	6.2500	82.00
15	0.1550	45.0000	6.2500	85.00
16	0.1550	45.0000	6.2500	84.00

- *Calculation of coefficients using coded variables*

The coefficients for the coded variables X_1, X_2, and X_3 were calculated using the NemrodW software, and the results are shown in Table 5. It is worth noting that the number of tests conducted was significantly greater than the number of coefficients to be calculated. Specifically, the model contains 10 coefficients, while the experimental plan involved 16 tests. This means that the number of degrees of freedom is equal to the difference between the number of tests and the number of coefficients, which is 16 - 10 = 6.

This information about the degrees of freedom is important because it helps to determine the statistical significance of the results. In this case, with six degrees of freedom, a statistical analysis can be performed using an appropriate distribution (such as the t-distribution) to determine whether the coefficients are 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 model applied is a multiple linear regression model, and its equation is as follows:

$$\mathbf{Y = 83.5 + 8.75\, X_1 - 5.34X_2 + 1.43X_3 - X_1^2 + 2.67X_2^2 + 1.03X_3^2 + 7.51X_1X_2 - 0.82X_1X_3 - 1.88X_2X_3}$$

In this model, Y represents the dependent variable (responses for inhibition efficiency), X_1, X_2, and X_3 are independent variables, and X_{12}, X_{22}, and X_{32} represent the squared terms of the respective independent variables. The other terms in the model are interaction terms between the independent variables. The coefficients associated with each variable and term in the model represent the impact of that variable or term on the dependent variable Y.

The standard deviation of the response, Se, is a measure of experimental errors that indicates the uncertainty of each test. Directly estimating Se allows for a better understanding of the precision of the data and the reliability of the results.

The standard deviation of each coefficient can also be calculated to determine if they are statistically different from 0. This is done by dividing the coefficient by its standard deviation and comparing the result to the values of a Student's t distribution. The fourth column in the output shows the standard deviation of the coefficient, and the next column shows the criterion t. The software used in the analysis provides the probability associated with the value of Student's t for a given number of degrees of freedom, which represents the risk α of being wrong by rejecting the hypothesis that the coefficient is zero.

In this particular study, the researchers have decided to consider a coefficient significant if the associated probability is less than or equal to 0.05 or 5%. This means that the researchers are willing to accept a 5% risk of being wrong by rejecting the hypothesis that the coefficient is zero. The significant coefficients in this study are b_1, b_2, and b12, which have associated probabilities less than or equal to 0.05.

In summary, the statistical analysis of experimental data involves calculating the standard deviation of the response and the standard deviation of each coefficient to determine the precision and reliability of the results. The significance of each coefficient is determined by comparing its value to the values of a Student's t distribution and calculating the associated probability, which represents the risk of being wrong by rejecting the hypothesis that the coefficient is zero.

The initial model ultimately simplifies to a simpler model, such as:

$$Y = 83.5 + 8.75X_1 – 5.34X_2 + 1.43X_3 + 7.51X_1X_2$$

The use of a regression variance analysis table in interpreting the results of a calculation program highlights the effect of the regression model compared to the residual effect and is used to assess the significance of the model.

The principle of the calculation involves decomposing the sum of the squares of the differences (SCE) into two components: the SCE due to the model and the residual SCE. The variances corresponding to these two sources of variation are then calculated and compared by a Fisher test. If the variance due to the regression is greater than the residual variance, we can conclude that the model is significant.

In other words, the regression variance analysis table provides a way to assess the overall fit of the regression model by comparing the variation explained by the model to 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 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, the regression variance analysis table provides a useful tool for interpreting the results of a regression analysis and assessing the significance of the model. By comparing the variance due to the regression to the residual variance, we can determine the overall fit of the model and make informed decisions about its use and application.

- *Analysis of variance*

Table 6 presents the results of the analysis for the obtained measurements. The calculated F-ratio corresponds to a F-distribution with 9 and 10 degrees of freedom, and the associated probability is less than 0.001. Therefore, it can be concluded that the selected model provides a statistically significant explanation for the 5% risk response variations.

Table 6. Variance analysis table

Source of variation	Sum of squares	Degrees de 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			

The important aspect of the analysis is to use the coefficient of determination to evaluate the performance of the chosen second-degree model. This measure gives the percentage of the total variance in the dependent variable Y that is explained by the model. In this case, the coefficient of determination is calculated to be 0.92, indicating that the chosen model can explain 92% of the variance in Y, which is considered a satisfactory result.

The estimation of the coefficients for the different models is shown in Table 5. However, it is not enough to know the estimated coefficients; we need to determine if they have a significant influence on the phenomenon being observed. To achieve this, the analysis uses a Student's t-test to determine if the estimated coefficients, denoted as bi, are significantly different from zero. If a coefficient is significantly different from zero, it suggests that it has a significant influence on the phenomenon being observed, and if not, it can be considered insignificant. Therefore, the Student's t-test is a crucial step in validating the impact of the different coefficients on the model's performance.

3.4. Optimal research and interpretation

The theoretical model finally preserved is:

$$\mathbf{Y = 83.5 + 8.75X_1 - 5.34X_2 + 1.43X_3 + 7.51X_1X_2}$$

We have validated the model we applied and can calculate the expected response at any point within the experimental area of interest. This knowledge can be visualized by plotting the calculated response values as points on a graph and connecting them to form isoresponse curves. These curves can be easily represented in two- or three-dimensional space.

However, the analysis is limited to those coefficients that have an effect on the inhibition efficiency.

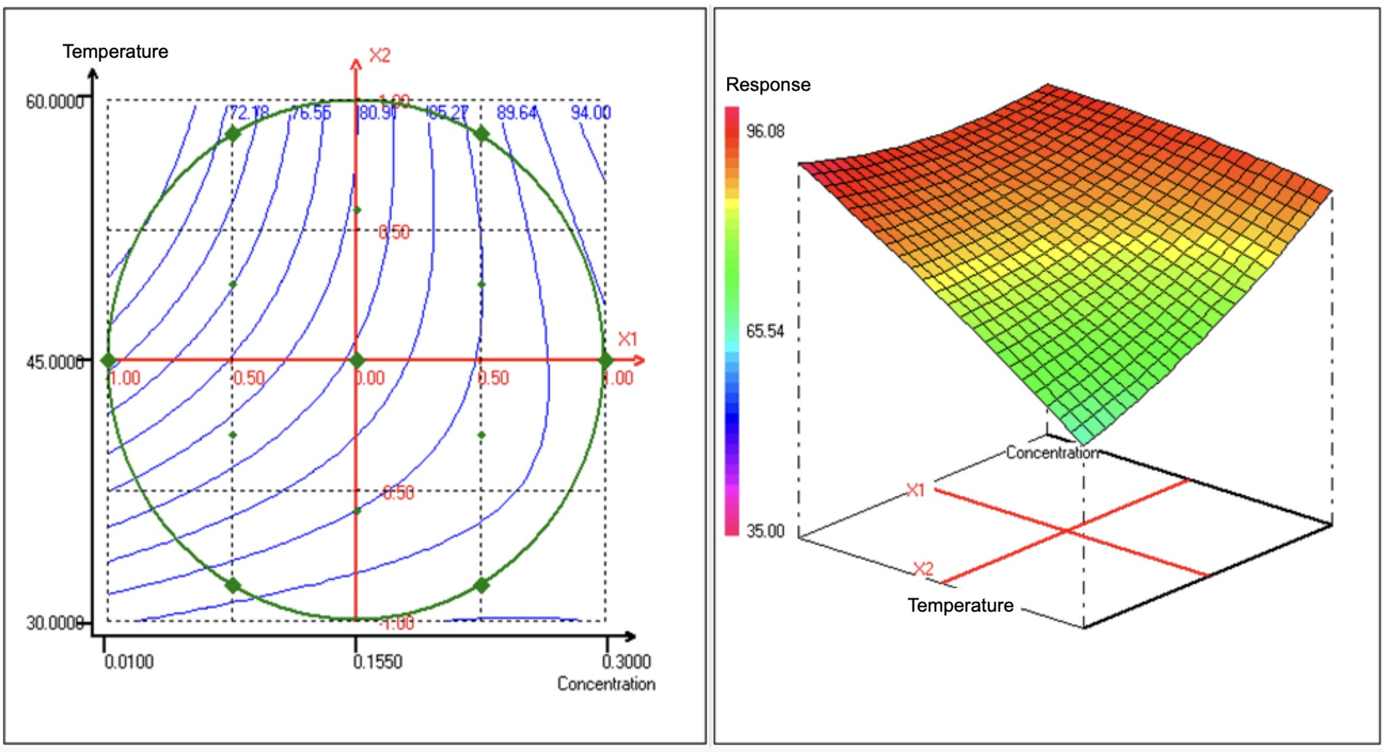


Figure 2. Variation in response - efficiency of the plan: Concentration, Temperature

FIXED FACTOR: - Time = 6.25 h

The iso-response curves, which plot inhibition efficiency as a function of inhibitor concentration and immersion time, provide a visual representation of this variation.

Furthermore, the observed decrease in inhibition efficiency with an increase in temperature suggests that higher temperatures may have a negative effect on the effectiveness of the inhibitor. This information could be useful in developing strategies to optimize the use of the inhibitor, for example by operating at lower temperatures to achieve higher inhibition efficiency. Overall, the results highlight the importance of carefully controlling both the inhibitor concentration and temperature in order to achieve optimal inhibition efficiency.

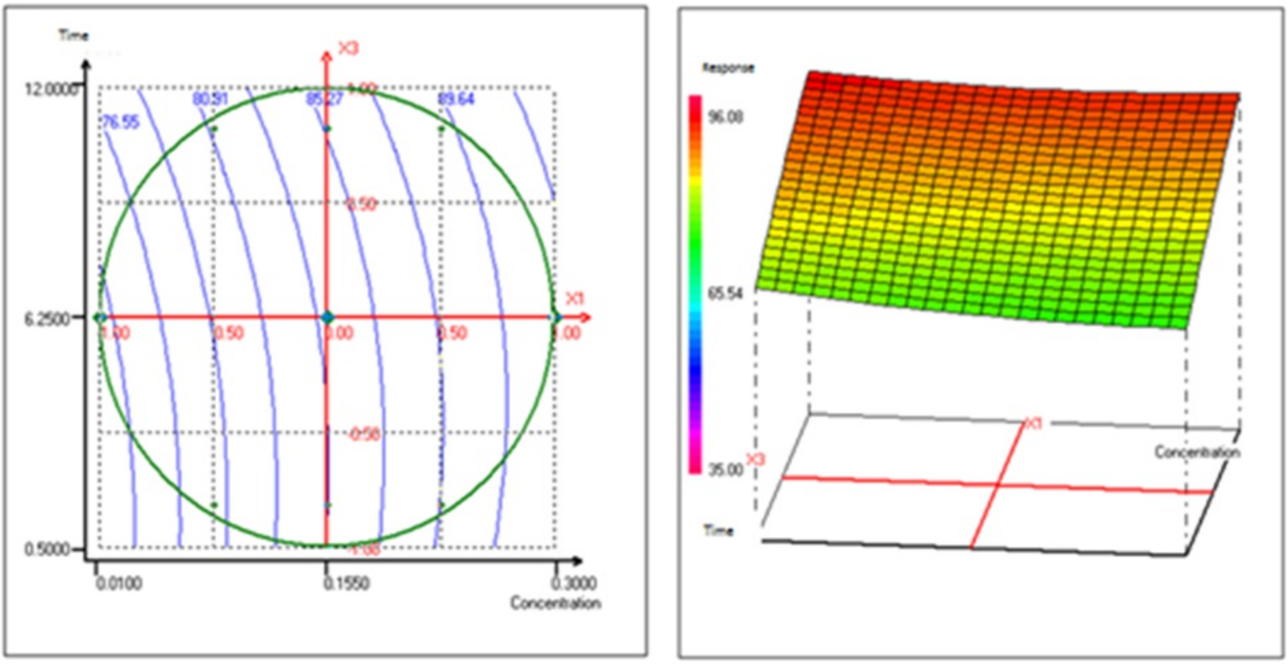


Figure 3. Variation in response - efficiency of the plan: Concentration, Time
FIXED FACTOR: - Temperature = 45 ° C

The results indicate that there is a variation in the inhibition efficiency when both the inhibitor concentration and immersion time are changed, while operating at a constant temperature of 40 °C. It was observed that, with an increase in immersion time, the inhibition efficiency also increased, indicating a positive correlation between the two.

This information can be useful in developing strategies to optimize the use of the inhibitor. For example, if it is desired to achieve a high level of inhibition efficiency, it may be beneficial to increase the immersion time while keeping the inhibitor concentration and temperature constant. This can lead to a more effective use of the inhibitor and better control over the system being inhibited.

Overall, the results suggest that carefully controlling the immersion time can be an important factor in achieving optimal inhibition efficiency, while also highlighting the need to consider the effects of other variables, such as inhibitor concentration and temperature, in the development of effective inhibition strategies.

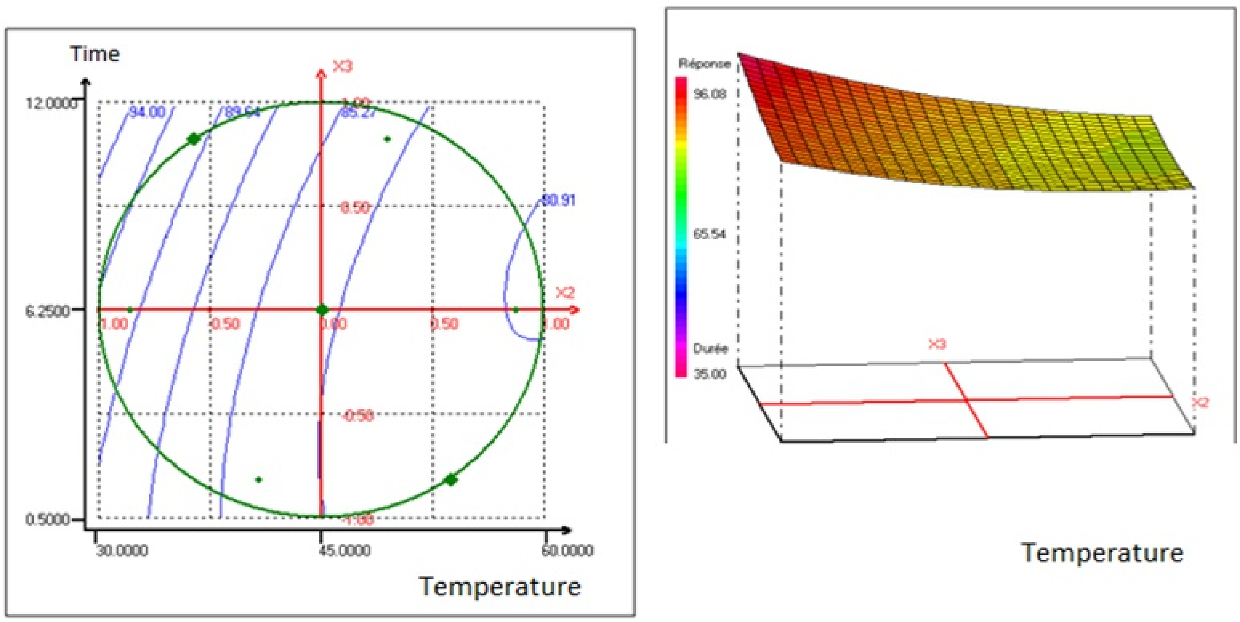


Figure 4. Variation in response - efficiency of the plan: Temperature, Time

FIXED FACTOR: - Concentration = 0.1550 mmol / l

The results indicate that there is a variation in inhibition efficiency when both the inhibitor concentration and immersion time are changed while operating at a constant temperature of 40°C. It was observed that with an increase in immersion time, inhibition efficiency also increased, indicating a positive correlation between the two.

This information can be useful in developing strategies to optimize the use of the inhibitor. For example, if a high level of inhibition efficiency is desired, it may be beneficial to increase immersion time while keeping the inhibitor concentration and temperature constant. This can lead to a more effective use of the inhibitor and better control over the system being inhibited.

Overall, the results suggest that carefully controlling immersion time can be an important factor in achieving optimal inhibition efficiency, while also highlighting the need to consider the effects of other variables, such as inhibitor concentration and temperature, in the development of effective inhibition strategies.

4. Conclusion

In conclusion, the experiment conducted showed that MDP is a highly effective inhibitor in 1M HCl. The experiment design methodology was carefully crafted to reduce the number of tests, saving time and resources. The mathematical model derived from the experiment is highly accurate and can be used to predict corrosion rates at any point within the experimental domain. Based on the data obtained, the optimum conditions for inhibition were found to be at the point with the coordinates (**Time=12h, Concentration of MDP=0.3 mmol/L, Temperature=30°C**). The methodology employed allowed us to determine the best operating conditions to achieve maximum inhibition efficiency of MDP, making this inhibitor a strong candidate for practical applications in the prevention of corrosion in acidic environments. Overall, the work provides valuable insights that can be applied in the real world to prevent corrosion and improve the durability of materials in acidic environments. The experiment design methodology was carefully crafted to save time and resources while ensuring the accuracy of the results.

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