Experimental Study and Response Surface Methodology of Azo Dye as an Eco-Friendly Corrosion Inhibitor for Stainless Steel in a Chloride Solution

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Abstract
DAD was investigated as CI for SS corrosion in a 3.5% NaCl solution, using WL, thermometric and RSM techniques. The analyses results show that IE(%) of DAD was dependent on its Ct and T. The highest IE(%) values were recorded on 5.0 g/L, at 303 K, by thermometric and WL methods (90.0 and 98%, respectively). DAD adsorption onto the SS surface was found to be spontaneous, obeyed Langmuir's isotherm, and its mechanism appeared to be partly physical, partly chemical. The results of RSM techniques supported experimental data. Finally, SEM was used to examine SS surfaces morphology without and with DAD, and its results confirmed the CI process.

Keywords: adsorption; corrosion; DAD; NaCl; RSM; SEM; SS; WL.

Introduction•

SS is a metallic material usually employed in many different fields, including architecture, building and chemical engineering, metals extraction, desalination and wastewater treatment facilities, oil and gas industry, transportation, aerospace, food and beverage sectors, due to its great corrosion resistance [1]. Cr in SS produces a passive film layer of Cr-rich oxide at lower T, in O presence, which is what gives the material its high resistance [1-2]. During SS maintenance procedures employed in most manufacturing industries, saline media is used to clean steel parts and remove rust and calcification, sometimes in acid and alkali presence [3]. The disadvantage of this essential procedure is alkali corrosive attack on the metal surface, which results in failings, or even long-term damage to machine parts.

• The abbreviations and symbols definition lists are in page 430.

CI are chemicals substances added to alkaline solutions to minimize corrosion. CI primary mechanism occurs through their molecule's adsorption onto the metal surface. Due to this process, water molecules are displaced, and a barrier that shields the metal surface from aggressive media is created. Thus, it is important to consider the variables that influence the adsorption level. The presence of heteroatoms and specific functional groups, and the availability of conjugated electrons are all factors that provide adsorption centres for inhibitor molecules to bind with the metal surface via physisorption and/or chemisorption processes. Organic compounds must meet these structural requirements to be effective CI. The solution T and the inhibitors Ct are other variables that significantly impact adsorption.

It is known that the substructures of several regularly used dyes are organic inhibitors and share significant similarities with most heterocyclic organic compounds like pyridines, furans, imidazoles, thiophenes and isoxazoles [3-7].

This characteristic has inspired scientists throughout the world to investigate the use of dyes as CI. Dyes have started to replace harmful CI that were previously used, because they are non-toxic and have a negative influence on the environment.

Azo dyes are amino based aromatic compounds. Literature has reported studies on the extensive use of amine-based aromatic compounds as CI for several metals and alloys in various electrolytes. Generally, the amino group ($-NH_2$) of such compounds serves as a site for interaction with the metallic surface, and the remaining molecules behave as water repellent [8].

[9] synthesized and characterized a novel coumarin azo dye as CI for MS in an acidic environment, applying both experimental and theoretical approaches. Their findings show that the dye inhibited the corrosion process and protected the MS surface. Table 1 lists other research on similar dyes.

Table 1: Works related to the present research on different metals and corrosive media.

Metals	Inhibitors	Corrosive media	IE(%)	Ref.
MS	Azo dye compound	HCl	96%	[18]
MS	[N-substituted p-amina azo benzene]	H_2SO_4	82.48%	[19]
Al	Mono azo dyes	NaOH	73%	[20]
MS	Mordant green 17	HCl	83.1%	[21]
CS	Benzonitrile azo dye	HCl	99.5%	[22]
CS	Azo chromoto tropic acid dye	H_2SO_4	82.3%	[23]
CS	Alizarin yellow dye	HCl	97.3%	[24]
MS	Allura red, sunset yellow and amaranth	H_2SO_4	90, 80 and 78%	[25]
MS	Chromotropic acid dye and	H_2SO_4	83 and 87%	[26]
SS	DAD	NaCl	90-98%	This study

In the present study, DAD was investigated as CI for SS immersed in NaCl. Fig. 1 depicts DAD molecular structure. In addition to its eco-friendliness and availability, the molecule is relatively bulky and rich in N heteroatoms, making it a promising adsorbate and, hence, a viable candidate for CI [10].

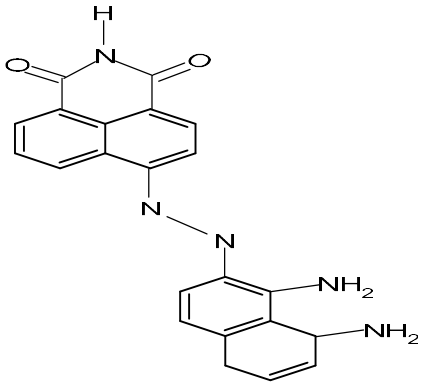


Figure 1: DAD molecular structure.

DAD molecular structure qualifies it as a suitable CI for two reasons: there are numerous N and O heteroatoms in its chemical structure, which have been widely discovered and proved in literature as excellent sites for adsorption onto the metal surface [11]. DAD has also a very high molecular weight. Literature has widely reported that IE(%) is higher as the molecular species mass increases. This phenomenon is often due to the inhibitor bulkiness, which increases the metal SC [11].

Corrosion and inhibiting processes have been investigated by several authors who have employed thermometric and RSM methods, which are vital in estimating and predicting the service life span of metals in real-world settings and are less time consuming [12-17]. However, DAD effects on SS corrosion behaviour are not yet well understood, and the use of RSM for predicting the inhibitor effect on SS in Cl media has not yet been reported until now.

Therefore, herein, experimental validation was done with the projected ideal process parameters, and the related IE(%) was examined using WL and thermometric methods, which are less expensive and quicker [10].

Experimental

Sample material and inhibitor preparation

In this study, a grade SS-410 SS sample with a thickness of 0.5 cm was employed, and it was cut into coupons of 5 x 1.5 cm. A 0.35 cm hole was drilled on each coupon. A twine was passed through this hole to aid suspension and total immersion in the media during WL measurements. SS-410 SS specimen chemical composition was: Cr (11.14%), Mn (0.82%), Cu (0.43%), C (0.134%), P (0.02%), Si (0.005%) and the remain Fe [2]. DAD was purchased from Sigma Aldrich chemicals and used without further purification. 3.5% NaCl was prepared and used for making the test solution with DAD. To prepare 5.0 g/L Ct of the inhibitor stock solution, 5.0 g/L DAD were dissolved in 1000 mL 3.5% NaCl solution. Then, the resulting solution was allowed to stand for 24 h, to enhance DAD solubility. Different Ct of DAD were calculated from the stock solution, using the dilution formula $C_1V_1 = C_2V_2$, and subsequently used in experimental measurements.

WL test

WL approach is the one that is most frequently used to evaluate CI, since it is quite easy and dependable. Many corrosion monitoring methods use WL as a foundational method. Herein, the SS surface was polished to a mirror shine using 1200 and 800 grit emery paper, then washed with distilled water, rinsed with acetone, and finally dried in a desiccator, before being submerged in the test solution (250 mL). WL experiment was carried out by SS total immersion in a 3.5% NaCl solution with and without DAD. The specimens were removed from the solution after 24 h, washed with a bristle brush under running tap water, for removing the corrosion product, dried in a desiccator, and reweighed precisely. WL of SS was determined by the difference between initial and final weights. For each solution, experiments were conducted thrice. The mean value was then recorded and used to calculate CR and SC [18-27].

Thermometric test

A calorimeter was used to perform thermometric test. The principle behind this technique is to monitor the change in T per min. This experiment was carried out at 30 and 60 ºC. IE(%) was evaluated from RN obtained in Eq. 1.

$$RN = \frac{T_m - T_i}{t} \quad (1)$$

where T_m and T_i are maximum and initial T in ºC, and t is the time taken in min to reach maximum T.

$$IE\% = \frac{RN_{Blank} - RN_{Inhi}}{RN_{Blank}} \times 100 \quad (2)$$

where RN_{blank} and RN_{inhi} are RN for SS corrosion in an aqueous solution without and with inhibitor, respectively.

RSM

For the data modelling and experimental runs, Design-Expert 13 software was employed. According to Table 2, the variables investigated were IT (A), T (B) and inhibitor Ct (C) (multiple input).

Table 2: CCD factor levels of independent variables.

Independent variable	Low factor level	Medium factor level	High factor level
A: IT (h)	24.0	72.0	120.0
B: T (ºC)	25.0	28.0	30.0
C: Ct of DAD (g/L)	0.1	2.6	5.0

Three levels of analysis were done on these 3 variables. To decide where the experimental run would take place, CCD was used in the trial design. 16 experimental runs were produced using CCD. The studies were conducted in random order, to prevent systematic errors. RSM results were assessed. The experimental evaluation of the various effects on CI at the design locations was carried out. A mathematical model was developed, for representing the relationship

between process factors and IE(%). Using RSM, one may predict the optimum value for the highest possible IE(%).
ANOVA and graphical analysis of IE(%) and CR were obtained from RSM, after responses evaluation (Table 3).

Table 3: ANOVA for CI of SS in 3. 5% NaCl with DAD.

ANOVA for RSM quadratic model [partial sum of squares - Type III]						
Source	**Sum of squares**	**Df**	**Mean square**	**F-value**	**P-value, Prob > F**	
Model	9814.19	9	1090.47	41.48	0.0001	Significant*
A-IT	3120	1	3120	15.1	1.0000	
B-T	8011.32	1	8011.32	304.71	<0.0001	
C- DAD	232	1	232	45.3	1.0000	
AB	18	1	18	2.7	1.0000	
AC	7	1	7	0.52	1.0000	
BC	4.3	1	4.3	0.38	1.0000	
A^2	15.26	1	15.26	0.5804	0.4750	
B^2	1511.65	1	1511.65	57.50	0.0003	
C^2	15.26	1	15.26	0.5804	0.4750	
Residual	157.75	6	26.29			
Lack of fit	157.75	5	31.55			
Pure error	0.0000	1	0.0000			
Cor total	9971.94	15				
STD				R^2		0.9842
Mean				Adj. R^2		0.9605
CV%				Pred. R^2		0.8802
Press				Adeq. precision		1194.58

It was also possible to derive mathematical models in terms of coded factors, which enabled to predict responses for a given level of each factor [28-30].

Results and discussion

WL measurement

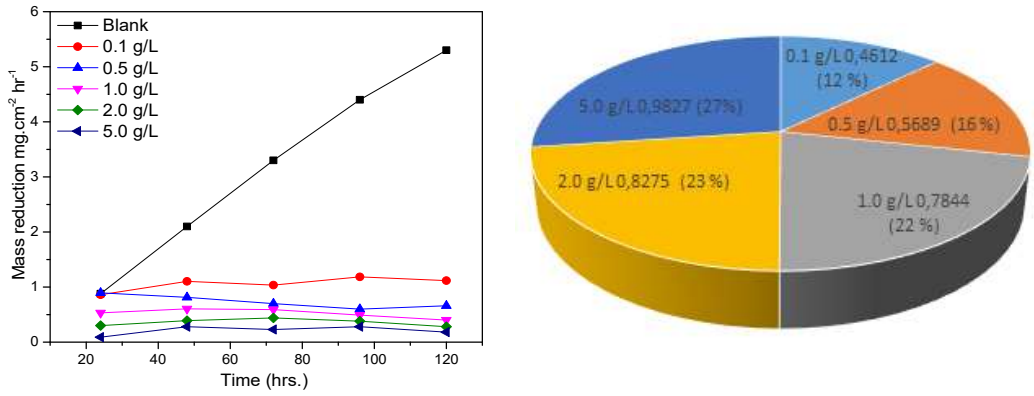
Fig. 2a displays calculated WL plots of SS in a 3.5% NaCl solution with and without various Ct of DAD, at 30 ºC. WL of SS decreased with higher Ct of DAD. This means that DAD inhibited SS corrosion in a NaCl solution. WL of SS was calculated by eq. (3).

$$\Delta M = \frac{m_1 - m_2}{At} \quad (3)$$

where m_1 and m_2 are WL of SS specimens before and after their immersion in NaCl. Eq. 4 was used to calculate SC on SS.

$$\theta = \frac{1 - CR_{inh}}{CR_{blank}} \quad (4)$$

where θ is SC of SS by DAD, and CR_{Inh} and CR_{blank} are CR values of SS without and with DAD, respectively. They were obtained from the slopes plotted in Figs. 2a and b. SC on SS by DAD in an aqueous medium depends on the inhibitor Ct, due to its adsorption onto the metal [31-32].

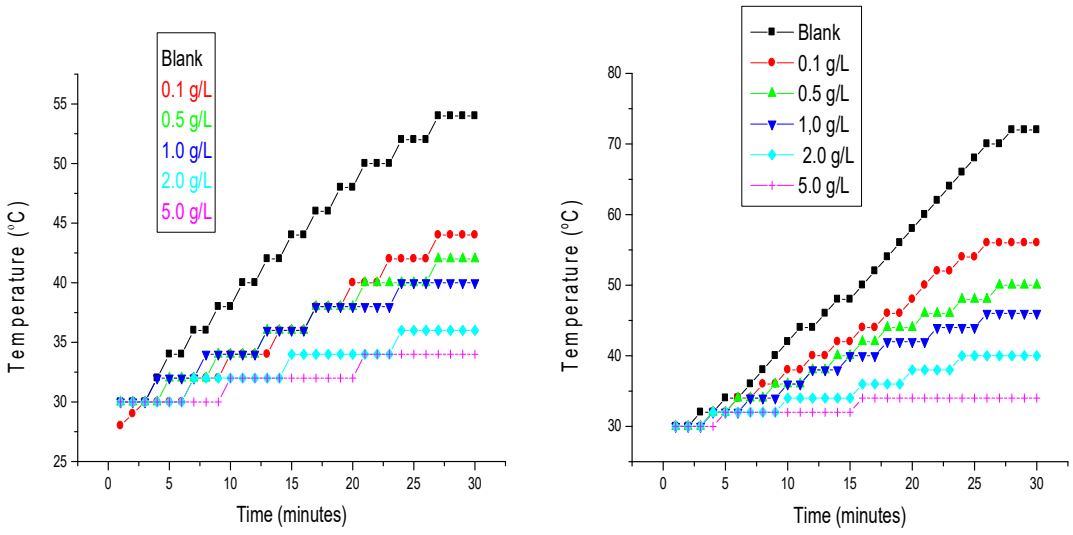


(a) **(b)**

Figure 2: (a) IT-WL curves of SS in a 3.5% NaCl solution with and without DAD, in various Ct, at 30 ºC; **(b)** chart showing SC of DAD on SS.

Thermometric measurement

Thermometric analysis has proven to be quite useful in the understanding of metal corrosion. The method can also be used to assess IE(%) of a variety of chemical substances. Figs. 3a and 3b depict T variation with IT, for SS corrosion in a 3.5% NaCl solution, without and with DAD, at various Ct. The system T rose progressively, due to the exothermic corrosion reaction. The blank solution RN, at 30 and 60 ºC, was recorded as 0.8 and 1.40 °C/min^{-1}, respectively. Table 4 also shows that RN values decreased with a higher Ct of DAD. This was due to DAD adsorption onto the SS surface, which prevented its corrosion in Cl solutions [27, 33].



(a) **(b)**

Figure 3: SS corrosion T-IT curves in 3.5% NaCl solutions without and with DAD, at various Ct.

Table 4: RN thermometric calculated values for SS corrosion in 3.5% NaCl with DAD.

Ct	T	RN	SC	IE(%)
blank		0.80		
0.1 g/L		0.53	0.337	33.7
0.5 g/L	30 ºC	0.40	0.500	50.0
1.0 g/L		0.33	0.587	58.7
2.0 g/L		0.20	0.750	75.0
5.0 g/L		0.13	0.837	83.7
blank		1.40		
0.1 g/L		0.93	0.385	38.5
0.5 g/L	60 ºC	0.66	0.528	52.8
1.0 g/L		0.53	0.621	62.1
2.0 g/L		0.40	0.764	76.4
5.0 g/L		0.20	0.907	90.7

Results of RSM analysis

Table 5 demonstrates how Ct, T and IT affected WL, CR and IE(%). An ideal IE(%) of 98% was attained.

Table 5: RSM results of SS corrosion in a 3.5% NaCl solution with DAD.

Std.	Run	Factor 1 (A) IT (h)	Factor 2 (B)T (ºC)	Factor 3 (C) Ct of DAD (g/L)	Response 1 WL (mg)	Response 2 CR ($mg/cm^2/h$)	Response 3 IE%
14	1	72	28	5	0.4	0.511	98
15	2	72	28	2.6	0.4	0.511	72
2	3	120	26	0.1	0.215	3.256	24
11	4	72	25	2.6	0.03	3.256	72
7	5	24	30	5	0.215	6	98
4	6	120	30	0.1	0.4	0.511	24
8	7	120	30	5	0.03	7.87	98
1	8	24	26	0.1	0.4	6	24
6	9	120	26	5	0.03	0.5	98
16	10	72	28	2.6	0.03	3.256	72
12	11	72	30	2.6	0.215	3.256	72
3	12	24	30	0.1	0.03	0.511	24
10	13	72	28	2.6	0.215	6	72
9	14	24	28	2.6	0.215	6	72
13	15	72	28	5	0.215	3.256	98
5	16	24	26	5	0.53	3.256	98

Predicted and actual IE(%) values are shown in Fig. 4a, which indicates a strong correlation between experimental and theoretical studies. Eq. 5 is the general quadratic model with significant and insignificant variables, such as IT (A), T (B) and inhibitor Ct (C), which links them to IE(%). The model was reduced to Eq. 6, when only significant terms were taken into consideration. According to the statistical study, there was only a 0.01% chance that F-value could occur due to noise, and Ct of O and flow rates of DAD functional groups would be uncontrollable

sources of noise. The models are important and useful for navigating the design environment [34].

$$\mathrm{IE}(\%) = +89.53 + 16\mathrm{A} + 25\mathrm{B} + 3\mathrm{C} - 5.1\mathrm{AB} - -5.0\mathrm{AC} - 5.6\mathrm{BC} - 1.28\mathrm{A}^2 - 12.77\mathrm{B}^2 - 1.28\mathrm{C}^2 \quad (5)$$

$$\mathrm{IE}(\%) = +89.53 + 16\mathrm{A} + 25\mathrm{B} + 3\mathrm{C} \; - 1.28\mathrm{A}^2 \quad (6)$$

RSM plots

Researchers were able to examine the interactive impact of process variables on the percentage IE(%), by plotting a three-dimensional surface curve against any two independent variables, while holding the other one constant. Fig. 4 describes predicted *vs.* experimental plots, demonstrating that they were properly distributed near the straight line. This indicates a strong relationship between experimental and predicted response values. It also confirms that the selected quadratic model could accurately predict response variables for experimental data. A linear graph showing the link between factors and DAD corrosion IE(%) response in the planned experiment was produced by predicted *vs.* experimental plots (Fig. 4). Figs. 5 to 7 illustrate the 3D surface plot.

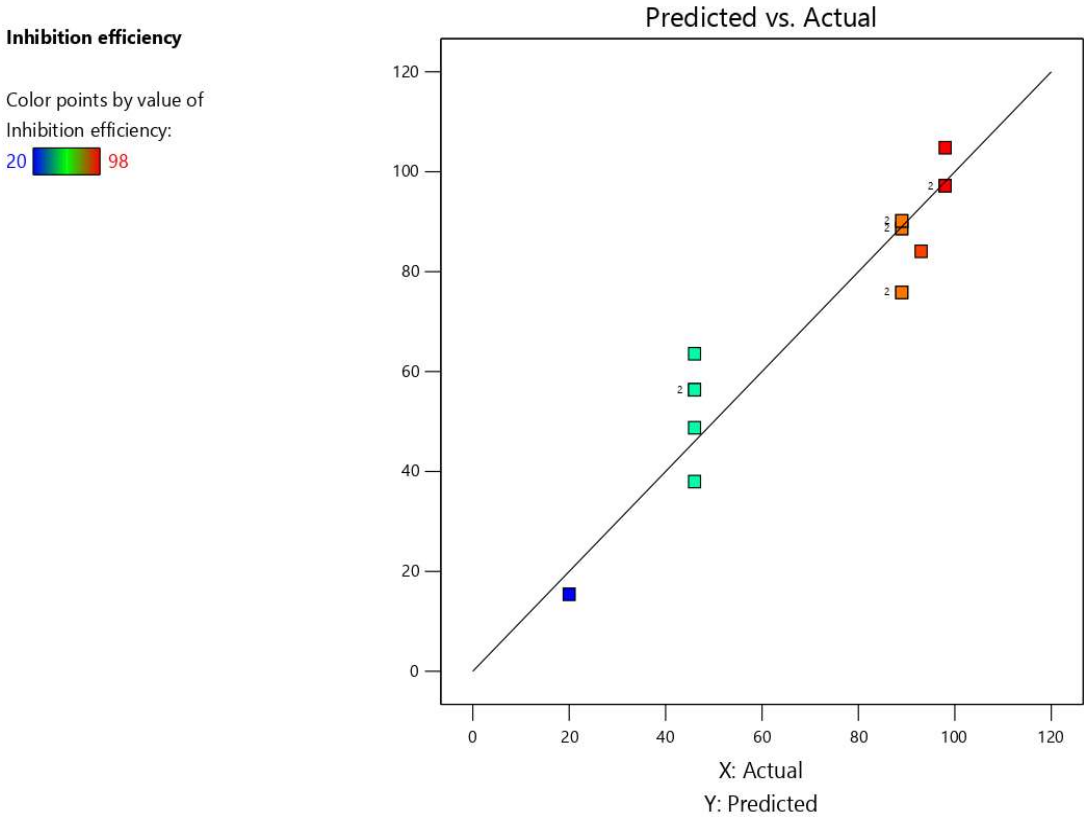


Figure 4: Plot comparing DAD predicted and experimental IE(%) values on SS corrosion in a 3.5% NaCl solution.

Fig. 5 shows that, with a given Ct of CI, IE(%) increases with IT, but decreases with higher T.

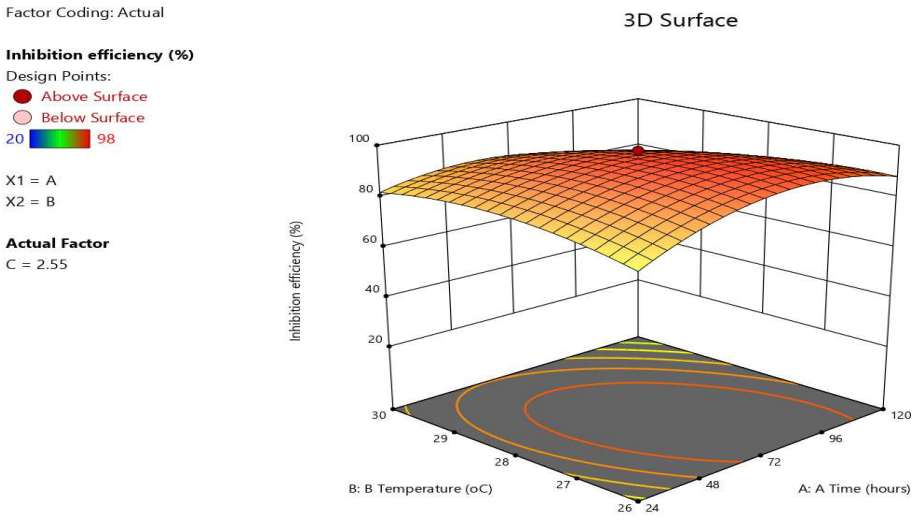


Figure 5: Effect of IT and T on DAD ability to inhibit SS corrosion in a 3.5% NaCl solution.

Fig. 6 reveals that CR decreased as Ct of DAD increased. However, CR increased with higher T. This confirms that the adsorption mechanism was physical, although Fig. 7 shows that WL decreased with higher Ct of DAD, and increased with prolonged IT [28-29, 34-35].

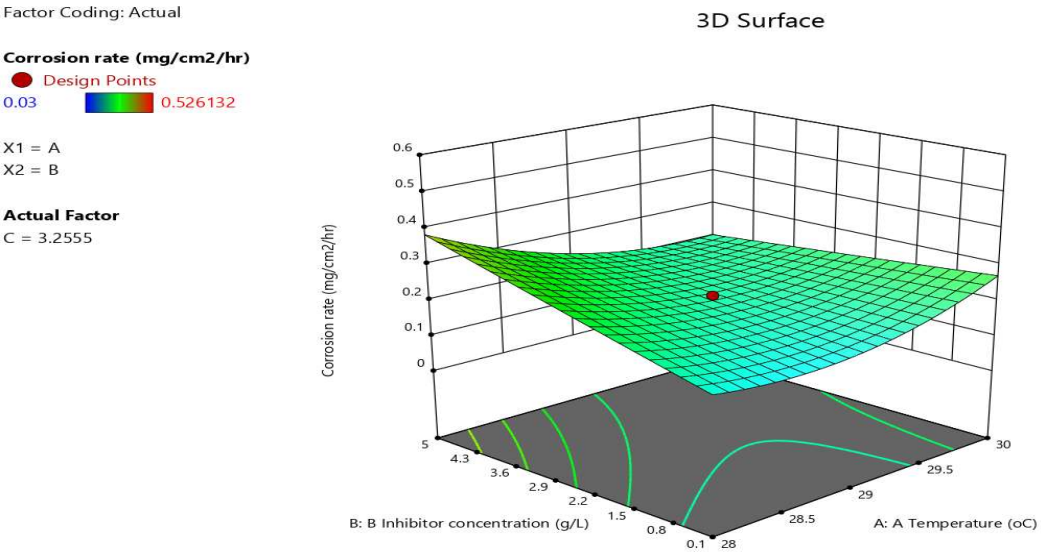


Figure 6: Effect of T and Ct of DAD on the CR of SS.

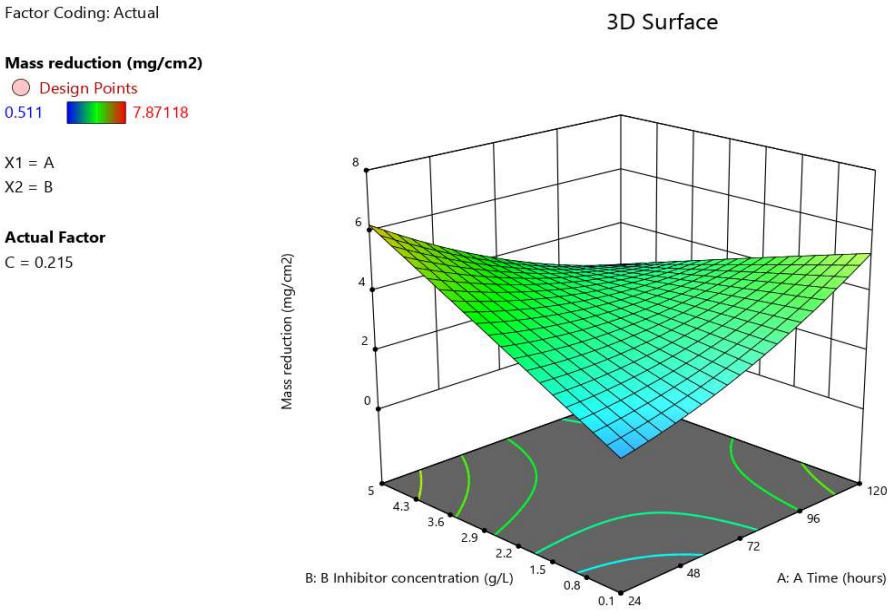


Figure 7: Effects of Ct of DAD and IT of SS on WL.

Adsorption isotherm

Langmuir's isotherm, which is defined by Eq. (7), is one of the most common methods for determining the adsorption nature of a CI.

$$C/\theta = 1/K_{ads} + C \qquad (7)$$

The plot of C/θ *vs.* C gave a linear plot with R^2 equal to 0.998 (Fig. 8).

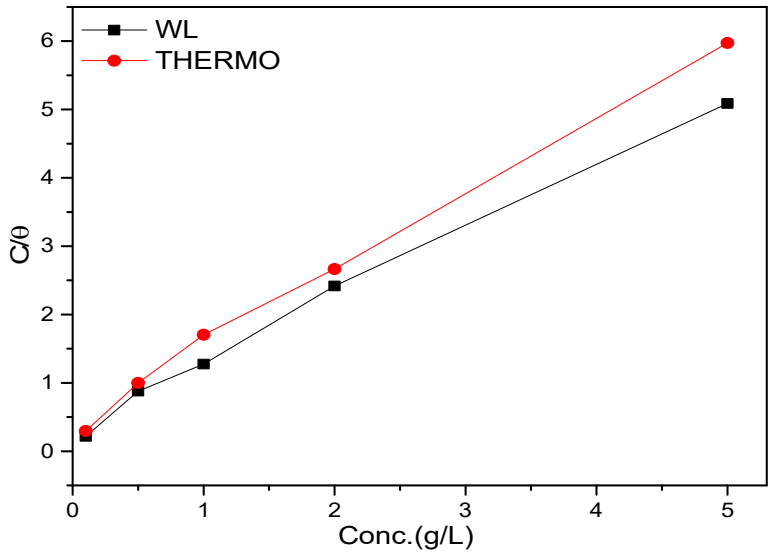


Figure 8: Langmuir's plot of SS in a 3.5% NaCl solution with DAD various Ct, at 303 K.

The plot suggests that DAD molecules were adsorbed onto the SS surface through a monolayer process, which is one of Langmuir's isotherm assumptions. ΔG^*_{ads} was calculated using Eq. 8.

$$\Delta G^*_{ads} = -RTIn(55.5K_{ads}) \qquad (8)$$

where R is the ideal gas constant, T is 303 K, and K is the inverse of the intercept of the linear plot on the y axis.
ΔG^*_{ads} calculated for WL and thermometric methods was -13.1 and -12.5 kJ/mol, respectively. The calculated values are negative and lower than the threshold values of 40 kJ/mol. This indicates that DAD adsorption mechanism was spontaneous and physical [36-42].

Surface characterization

After 24 h of IT, the SS surface was assessed without and with DAD (5.0 g/L) using SEM technique. It is clearly seen on SEM images (Fig. 9a) that the SS surface was seriously affected in NaCl without DAD. However, with the CI, it was only slightly damaged (Fig. 9b). This revealed the formation of a film on the SS surface (via DAD molecules adsorption) [43-56].

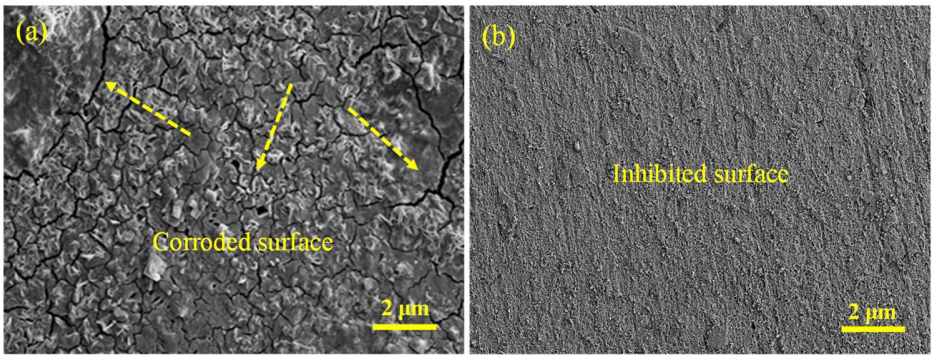


Figure 9: SEM images of SS **(a)** in NaCl without and **(b)** with DAD.

Conclusions

In this study, DAD was tested as CI of SS in a 3.5% NaCl solution. From the obtained results, the following conclusions were taken: experimental analyses indicated that DAD acted as efficient CI for SS in a 3.5% NaCl solution; thermometric measurements revealed that IE(%) increased with DAD higher Ct, at raised T; DAD adsorption onto the SS surface obeyed Langmuir's isotherm, and the mechanism was partly physical and chemical; RSM predicted CI of SS by DAD in a 3.5% NaCl solution. Error analysis revealed RSM technique superiority in modeling the CI of SS. Results from the significance test for model coefficients indicated that Ct of Cl was the most important factor in SS corrosion process; and SEM analysis showed that DAD, in the optimum Ct of 5.0 g/L, at 30 ºC, caused a significant reduction in the surface damage caused by the Cl solution attack.

Author's tasks

Fidelis Ebunta Abeng: conceived and designed the research work; performed experimental work; wrote the draft; conducted the review before submission.

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Abbreviations

ANOVA: analysis of variance
CCD: central composite design
CI: corrosion inhibition/inhibitor
Cl: chloride
CR: corrosion rate
CS: carbon steel
CV: coefficient of variation
MS: mild steel
Ct: concentration
DAD: Diaminonaphthalene azo dye
HCl: hydrochloric acid
H_2SO_4: sulfuric acid
IE(%): inhibition efficiency
IT: immersion time
MS: mild steel
NaCl: sodium chloride
NaOH: sodium hydroxide
R^2: correlation coefficient
RN: reaction number
RSM: response surface methodology
SC: surface coverage (θ)
SEM: scanning electron microscopy
SS: stainless steel
STD: standard deviation
T: temperature
WL: weight loss

Symbols definition

ΔG_{ads}: adsorption free energy

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