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Optimization of Mild Steel Corrosion Inhibition Using Expired Ciprofloxacin with Eggshell Powder

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Abstract- This return to the native oxide state is known as corrosion and even though it is inevitable, substantial barriers (corrosion control methods) can be used to slow its progress toward the equilibrium state. This study optimized the reaction conditions that could influence inhibition of mild steel corrosion in acidic medium using expired antibiotic ciprofloxacin doped with calcium rich egg shell wastes. four-factors-five-levels were optimized within operational corrosion parameters; acid concentration (AC) 1–2.5 M, immersion time (IT) 1–4 hr, inhibitor concentration (IC) 100–400 mg/L, and temperature (T) 303–343K. Twenty-four (24) independent experiments were conducted and results analyzed using analysis of variance. The model generated F-values and p-values were 7.03 and 0.02 for W, 4.67 and 0.04 for CR, 2.99 and 0.04 for %IE respectively with R^2 above 70%. Influence of reaction parameters was also studied as they influence the corrosion factors. The inhibitor was able to reduce the corrosion of mild steel in HCl medium.

Keywords: Optimization, Corrosion, Mild Steel, Antibiotic, Egg Shell.

I. INTRODUCTION

The development of modern society and industry has led to a stronger demand for engineers with specialized knowledge in corrosion (Yamin et al., 2020; Emembolu, et al., 2022). Mild steel is a solid substance obtained from the earth. It is a general-purpose engineering construction material mostly used as a result of its natural properties like rigidity, ductility, tensile strength conduct of heat and electricity etc (Jomy & Prabhu, 2025). Mild steel has numerous industrial applications and is mainly used for the corrosion protection of steel. Generally, corrosion resistance of metals such as steel, in corrosive environment, may be attributed to the formation of a protective tightly adhered invisible oxide film on the metal surface (Shwetha et al. 2024; Li et al., 2024). It is used for the production of lightly stressed machine fittings, shafts, turbine motors, railways axels, crank shafts, and fish plates, cross heads, pipes and drums. Although, the economic importance of these metals are hindered by corrosion activities (Balangao, 2024). Hence the corrosion of metals is often considered as an inconvenience because it implies a change of the objects/metals in the course of time. Owing to the increasing ecological awareness, as well as the strict environmental regulations, and consequently the need to develop environmentally friendly processes, attention is currently focused on the development of "green" alternatives to mitigating corrosion. Green approaches to corrosion mitigation entail the use of substances, techniques, and methodologies that reduce or eliminate the use of products, by-products, solvents, reagents, and so forth that are hazardous to human health or the environment in combating corrosion (Chen et al., 2024; Emmanuel, 2024; Li et al., 2024). Due to the appearance of heteroatoms such as N, O, P, Cl, and S, which demonstrate their inhibitory efficacy, some researchers have employed a variety of expired antibiotics as corrosion inhibitors. Expired Ambroxol (Srinivasulu & Kasthuri, 2017) expired Ceftazidime (Kamel et al., 2021), expired Ampicillin - Flucloxacillin (Alfakeer et al., 2020).

II. MATERIALS AND METHODS

2.1 Preparation of Acid Solution

The solutions of 1M HCl and 1M H_2SO_4were separately prepared using distilled water. The solutions were prepared with the aid of electronic weighing balance, volumetric flask and beakers. 85.91 ml HCl (36% pure, specific gravity of 1.18) was added to 600 ml of distilled water in 1liter measuring cylinder. The solution was made up to 1 liter with addition of distilled water. For the preparation of 1M H_2SO_4, 54.35 ml H_2SO_4 (98% pure, specific gravity of 1.84) was added to 700 ml of distilled water in 1 liter measuring cylinder. The solution was made up to 1 liter with addition of distilled water.

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2.2 Preparation of inhibitor

Exactly, 1M of ECPF inhibitor was prepared by dissolving ...g of ECPF in 1 Litre solution of distilled water, serial dilution was then used to obtain 100-500mg/L concentrations.

2.3 Metals Preparation

The mild steel metals were cut into coupons; 5cm x 4cm x 0.1cm. The coupons were cleaned with acetone followed by polishing with emery paper to expose shining polished surface and also remove any oil and organic impurities, the coupons were degreased with acetone and finally washed with distilled water, dried in air and then stored in desiccators. Accurate weight of each coupon was taken using electronic weighing balance and the initial weight was recorded. The coupons were labeled in a manner to avoid any mix up.

2.4 Scanning Electron Microscopy (SEM) analysis of metals

Scanning electron microscopy (SEM) was used for monitoring the surface morphological changes. In this study, polished aluminum and mild steel coupons were immersed in 1 M HCL, H_2SO_4, KOH and NaOH media respectively in the absence and presence of the 0.2- 1.0g/l of the inhibitor for 8hrs. Then the specimen was cleaned with distilled water, dried in acetone and used for the analysis.

2.5 CCD experimental study

In order to optimize and forecast the corrosion inhibition of mild steel in various HCl concentrations using thermometric and gravimetric methodologies, the RSM approach is applied. Reaction number (RN), weight loss (Δw), corrosion rate (CR), and percentage inhibition efficiency (%IE) are the responses to be employed for thermometric and gravimetric techniques as shown in Table 1. The experimental design (Table 1) consists of four-factors-five-levels array optimized within the following operational corrosion parameters; acid concentration (AC) 1–2.5 M, immersion time (IT) 1–4 hr, inhibitor concentration (IC) 100–400 mg/L, and temperature (T) 303–343K. Following the choice of the design template, twentyfour (24) independent experimental interactions will be conducted in duplicates according to the designated interactions for the control corrosion matrix for both thermometric and gravimetric methodologies.

III. RESULTS AND DISCUSSION

Table 1. Design of experiment for corrosion studies

Std	Run	AC (M)	IC (mg/L)	Time (hr)	Temp (K)	W (g)	CR (mg/cm 2hr^{-1})	I.E (%)
20	1	2.5	250	4	323	0.5044	0.3866	55.5
14	2	1.75	400	1	323	0.5096	0.2113	62.15
1	3	1	100	2.5	323	0.508	0.2681	60.31
10	4	2.5	250	2.5	303	0.5276	0.2774	53.48
11	5	1	250	2.5	343	0.5182	0.273	53.93
19	6	1	250	4	323	0.4855	0.2574	57.13
2	7	2.5	100	2.5	323	0.4846	0.257	57.24
6	8	1.75	250	4	303	0.5166	0.2137	61.88
23	9	1.75	100	2.5	343	0.5075	0.2679	60.18
15	10	1.75	100	4	323	0.5216	0.2155	64.3
9	11	1	250	2.5	303	0.5076	0.389	65.29
12	12	2.5	250	2.5	343	0.5066	0.2674	62.31
7	13	1.75	250	1	343	0.4935	0.2056	56.44
5	14	1.75	250	1	303	0.4816	0.2555	57.02
8	15	1.75	250	4	343	0.5091	0.2686	54.62
21	16	1.75	100	2.5	303	0.5078	0.3891	54.87
24	17	1.75	400	2.5	343	0.5287	0.4046	52.88
3	18	1	400	2.5	323	0.5076	0.389	54.75
13	19	1.75	100	1	323	0.4464	0.3436	60.52
4	20	2.5	400	2.5	323	0.4756	0.2527	57.57
17	21	1	250	1	323	0.4267	0.1822	61.95
18	22	2.5	250	1	323	0.5244	0.4014	60.88
22	23	1.75	400	2.5	303	0.5075	0.2679	55.24
16	24	1.75	400	4	323	0.5074	0.3888	62.1

Model equations:

$$W = +0.5269 +0.0021A+0.0087B+0.0154C+0.0032D-0.0021AB-0.0197AC-0.0079AD-0.0194BC+0.0054BD-0.0048CD-0.0169A^2-0.0125B^2-0.0232C^2-0.0111A^2B-0.0057A^2C-0.0160AB^2+0.0270AC^2 \quad (1)$$

$$CR = +0.02545-0.0293A+0.0165B+0.0088C-0.0088D-0.0313AB-0.0225AC +0.0265AD +0.0764BC +0.0645BD +0.0262CD+0.0591A^2+0.0659B^2-0.0187 C^2+0.0063$$

$$A^2C-0.0076AB^2+0.1164AC^2-0.0063BC^2-0.0878A^2B^2 \quad (2)$$

$$I.E=+51.02-0.5317A-1.06B-0.2858C \; 0.6183D+1.47AB+5.05AD-0.9575BC-1.92BD +7.74A^2 +4.78B^2 +6.47C^2-6.06 \; A^2B^2-6.36 \; A^2C^2 \quad (3)$$

3.1 Weight loss

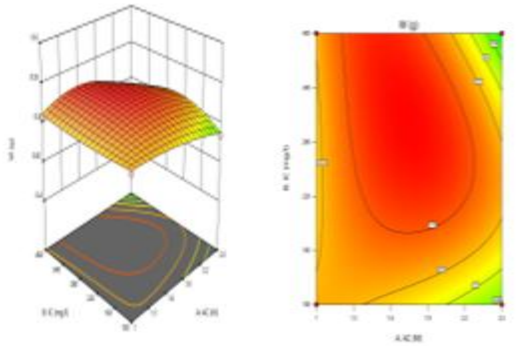

Figure 1. Plot of interaction for weight loss

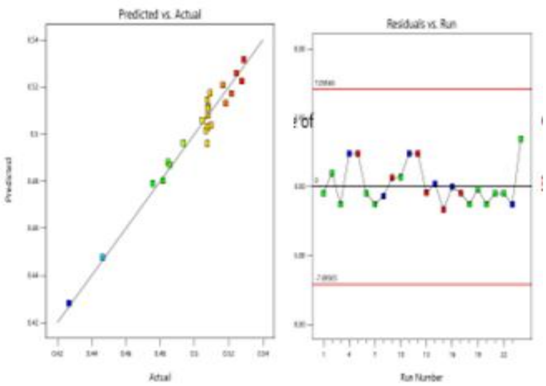

Figure 2. Diagnostic plot for weight loss

3.1 Influence of operational parameters

The design of experiments for corrossion inhibition of mild steel is shown in Table1. RSM was used in conjunction with Box behnkett approach for the optimization of the four factors acid concentration (AC), inhibitor concentration (IC), time (hr) and temp (K) process. The 3-D plots shown in figure 1a,3a and 5a respectivey. For weight loss (W), corrossion rate (CR) and inhibitor efficiency (%IE) demostrates that as inhibitor concentration also increases, further increasing acid concentration does not result in an increase in weight loss because a protective film emerges on the mild steel preventing HCl penetration. Moreover, an increase in inhibitor concentration increases the percentage inhibition efficiency. Also, contour plots were employed to explain the degree to which the operational factors interacted with their responses. As demonstrated in Figure 1b. an elliptic contour denotes an interactive effect and a circular contour illustrates a non-interactive effect. The circular plots indicates a non-intearctive effect of the paramenters on the responses. Consequently, two diagnostic tools; the plot of predicted against actual, and residual versus run were adopted to validate the developed RSM models as illustrated in Figure 2a,4a and 6a. respectively. The plot of prdicted against actual values show the convergence of experimental and RSM predicted data to the line of best fit while the plot of predicted versus run illustrates that there is no outlier, that is no value outside the red lines, all residual values are within the significant region, and correspond closely to zero residual error based on the difference between experimental and predicted values. This compliance demonstrates the RSM models' accurate prediction and the profound correlation between the predicted and experimental results.

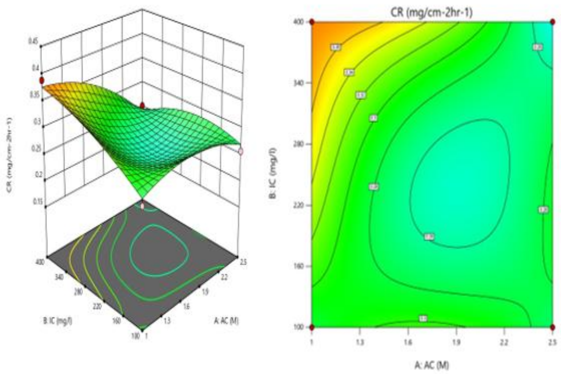

Figure 3. Plot of interaction for corrosion rate

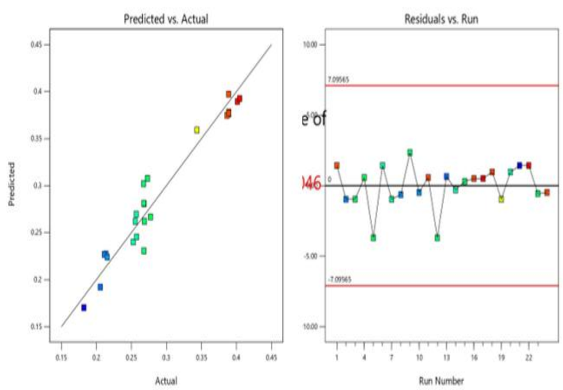

Figure 4. Diagnostic plot for corrosion rate

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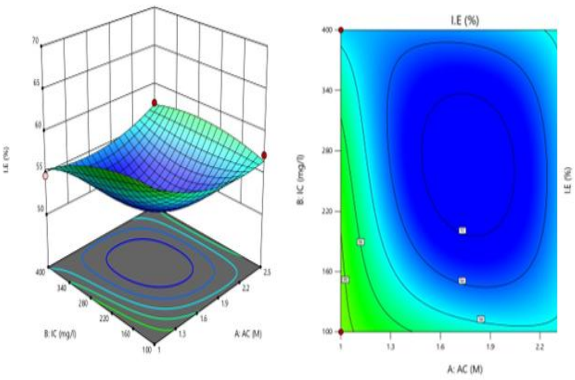

Figure 5. Plot of interaction for inhibition efficiency

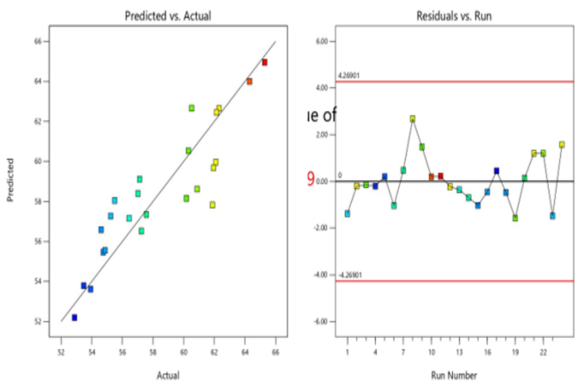

Figure 6. Diagnostic plot for inhibition efficiency

Table 2. ANOVA Summary

Factor	Source	Sum of Squares	df	Mean Square	F-value	p-value	
W	Model	0.0130	18	0.0007	7.03	0.0201	significant
CR	Model	0.1139	18	0.0063	4.67	0.0479	significant
% IE	Model	246.56	13	18.97	2.99	0.0448	significant

3.2 Development and validation of RSM models
The level of the correlation between the experimental and predicted values is defined by the analysis of variance (ANOVA) as shown in Table 2. The prediction model equation is considered to be adequate when the F-value is high, while the significance of the prediction model is obtained when p-values are low. The models used for prediction of corrosion factors W, CR and %IE all have P-values less than 0.05 which indicates that the prediction models are significant. The model generated F-values and p-values were 7.03 and 0.02 for W, 4.67 and 0.04 for CR, 2.99 and 0.04 for %IE respectively. The high Model F-values imply the models are significant and there is only a little chance that an F-value this large could occur due to noise. The correlation regression (R^2) for the variables indicates the degree of agreement between experimental results and theoretical predictions and was obtained as 0.9620 for W, 0.9439 for CR and 0.7954 for % IE in table 5. This suggests that the models are fit and good for analysis. The predicted and adjusted R^2 a less than 0.2 difference as required. Adeq Precision measures the signal to noise ratio, the ratio of greater than 4 that was obtained in all analysis is desirable. Therefore, the models used in equations 1-3 were suitable to navigate the design space.

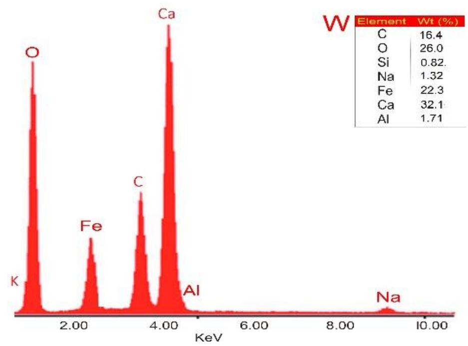

Figure 7. EDX of eggshell powder

3.3 EDX of eggshell powder
The electron dispersive X-ray of eggshell powder is shown in Figure 7. And denotes the presence of calcites and alumino silicates with alkali metals. These are suitable to promote corrosion inhibition of mild steel.

IV. CONCLUSION

- The study suggests that the ciprofloxacin was able to inhibit and reduce the corrosion rate of mild steel in HCl medium at the various reaction conditions in optimization studies.
- No Interaction was observed between the acid medium and inhibitor concentration in reducing the corrosion rate for all the studies.
- Increasing inhibitor concentration favored corrosion rate and weight loss but was not favored for inhibition efficiency,
- While diagnostic plots to study the efficiency of the optimization process confirmed a close relationship between the experimental results and theoretical model predictions with no outlier results in all studies.

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