



Anti-corrosion using rice straw extract for mild steel in 1.5 M H₂SO₄ solution

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ABSTRACT

Recent research has focused on use of green inhibitors to combat corrosion in industries. This study investigates the effectiveness of rice straw extract (RSE) on mild steel corrosion mitigation in 1.5 M H₂SO₄ using an optimization technique. Temperature (30–60 °C), inhibitor concentration of 0.2–0.8 g/l and time (3–7 h), were the variables. Phytochemical analysis was used to investigate the chemical constituents of RSE. Scanning electron microscopy was used for the characterization. The thermometry test's maximum inhibitory efficiency was 85.11%. The experimental design revealed a maximum inhibition effectiveness of 84.71%, but the verified experiment revealed a maximum inhibition efficiency of 86.42%. The result of the SEM showed that more adsorption was observed on mild steel of the validated experiment due to the bioactive constituents of the extract.

1. Introduction

Corrosion inhibitors are the cheapest approach to increase the useful life of mild steel equipment [1]. The greatest method for defending against damage on metals is to use plant extracts as corrosion inhibitors (green technology) [2]. Phytochemical factors like tannins, alkaloids, amino acids, flavonoids, phenols, saponins, are characteristics responsible for a good corrosion inhibitor. Some of the plant extracts reported as good inhibitors in the literature are: *Cyperus rotundus* [3]; Chamomile flower extract [4]; *Nepta Pogonesperma* plant stems extract [5]; *Chrysanthemum indicum* extract [6]; *dactylifera* extract [7]; friendly roselle (*Hibiscus Sabdariffa*) leaf extract [8]; Terebinth extracts [9]; Extract of *Cocos nucifera* - Coconut Palm [10]; *Oxalis stricta* leaf extract [11]; *Corchorus olitorium* stem [12]; *Hunteria umbellata* seed husk extracts [13]; *Tagetes erecta* [14]; Pawpaw leaves [15]; *Centipeda minima* leaves extract [16]; *Luffa cylindrica* [17]; *Xanthium Strumarium* leaves extract [18]; *Rollinia occidentalis* [19]; cashew extract [20]; *tribulus terrestris* plant [21]; licorice plant extract [22]; *Phyllanthus niruri* [23]; Cashew Nutshell [24]; *Lavandula* and *Ricinus communis* oil [25]; Parsley (*Petroselinum sativum*) extract [26]; *Aizoon canariense* Extract [27]; *Senecio anteuphorbium* [28]; *Aesculus hippocastanum* seeds extract [29]; *Rosa canina* fruit extract [30]; Bark Extract of *Tamarix aphylla* [31]; *Primula vulgaris* flower aqueous extract [32]. The gap in this study is the use of

rice straw which is an agricultural waste as corrosion inhibitor on mild steel using optimization approach. No work had been done on the use of Box Behnken Design for optimization as at when this research was done. This work is to examine in novelty, rice straw's corrosion-inhibiting efficiency.

2. Materials and method

2.1. Preparation of rice straw extract (RSE)

Rice Straw (RS) obtained from Landmark University Commercial Farm was dried for five (5) days and pulverized. 40 g of the RS powder was immersed in 1000 ml of ethanol for 48 h. After that, it was filtered at a lower pressure. A tiny amount of distilled water was used twice to remove the solid residue. A desiccator was used to store the product after the filtrate had been evaporated and concentrated at 80 °C.

2.2. Metal preparation

The mild steel used has the following element compositions (in weight percent): 0.15 (C), 0.15 (Si), 0.49 (Mn), 0.05 (P), 0.062 (S), and 0.05 (Cr).

To ensure complete immersion in the corrosive liquid, experiments

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were conducted using rectangular mild steel coupons with 0.1 mm diameter holes that were mechanically press cut into the 19 mm × 19 mm × 2 mm coupons. To get a smooth surface and remove any dirt from the metal surface, samples were abraded using emery paper.

After that, acetone and ethanol were used to clean and degrease. Then, in order to shield them from the effects of moisture, they were allowed to dry and kept in a desiccator.

2.3. Preparation of corrosive medium

The, solution in the concentration range of 0.2–0.8 g/l were utilized in the corrosive medium of 1.5 Molar H₂SO₄. The total amount of RSE used for this study was 200 g/l.

2.4. Thermometric test

Thermometric test was conducted using 50 ml beaker with inhibition solution and mild steel in the thermometric bath at 30 °C. Temperature changes were noted until steady temperature was observed. Equation (1) was used to calculate the reaction number (RN). Methodology adapted from Ref. [33].

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

where T_m and T_i are the final and initial temperatures in degrees Celsius, respectively.

t is the time.

2.5. Phytochemical analysis

The RSE was subjected to phytochemical investigation to check if bioactive constituents were present.

2.6. wt loss measurement

Studies of weight loss were conducted on pre-weighted coupons submerged in 250 ml volume beakers containing 100 ml inhibitor solution held at 30–60 °C in a water bath with thermostat. The coupons were put in solution at the time variation of 3–7 h after which they were re-weighted. Difference in weight was considered as weight loss.

The corrosion rate (CR), inhibition efficiency (IE), and the surface coverage were calculated using Eqs. (2)–(5).

$$\Delta w = w_1 - w_2 \quad (2)$$

Weight before immersion is denoted by w₁ and weight after immersion is denoted by w₂.

$$CR = \frac{\Delta w}{At} \quad (3)$$

where CR is the corrosion rate (g/mm²h).

Following an exposure time t (h), Δw is the weight loss (g), and A is the area.

Inhibition efficiency (IE) was calculated using Equation (4).

$$IE = \frac{\Delta w}{W_1} \times 100 \quad (4)$$

The surface coverage (Θ) was calculated using Equation (5).

$$\Theta = \left(1 - \frac{\Delta w}{W_1}\right) \quad (5)$$

The methodology used was adapted from Ref. [17].

2.7. Design of experiment

For the 17 developed experimental runs, the effect of variables was

examined using the Box Behnken Design. Table 1 showed the variables, while Table 2 showed variable interaction matrix. The Design Expert software (6.0.8) was used for analysis.

2.8. Optimal predicted levels by the software

Time: Time: 5 h, Temperature: 60 °C; Concentration: 0.40 g/l was observed by the software as the optimal process level.

2.9. Surface analysis

The surface analysis was done for coupon with highest inhibition efficiency (from experimental design), coupon of the optimal process level (validated), and blank coupon.

3. Results and discussions

3.1. Result of thermometric test

The result of Thermometric Test is as shown in Table 3. It was found that as the Inhibitor Concentration increased, the reaction number decreased. The highest Inhibition efficiency observed was 85.11%. at 0.8 g/l. This result is higher than what was observed by [20].

3.2. Result of phytochemical analysis

Table 4 showed result of phytochemical analysis. It was observed that RSE contained bioactive constituents of a good inhibitive action of the extract.

3.3. Results of weight loss measurement

Table 5 showed results of weight loss measurement. The 10th Experimental Run's best process level; with: 5 h 60 °C 0.40 g/l as shown in Table 6 with the highest IE of 84.71%.

3.4. Result of the statistical analysis and optimization study

Table 6 showed the ANOVA. The coefficients were used to determine model's goodness (R² and adjusted R²). The observed R² was 0.7838, while the adjusted R² was 0.5676. Analysis of ANOVA for the corrosion inhibition is as shown in Table 6. Fig. 1 showed the relationship of Predicted vs actual plot. The significant terms are B², C², AB. The regression equation is as stated in equation (6):

The temperature and the H₂SO₄ concentration is correlated with the IE.

Regression Equation:

$$IE = +71.68 - 0.57A - 1.74B + 0.81C - 1.47A^2 + 4.19B^2 - 25.80C^2 - 6.76AB + 2.60BC \quad (6)$$

The mechanistic interpretation revealed that the inhibitor concentration (C) had the greatest impact on the IE (response), with the effects of temperature (A) and H₂SO₄ concentration (B) having a substantially lower impact.

Table 1
Independent variables.

Independent variables	Codes	Range and levels		
		-1	0	+1
time (h)	A	3	5	7
temperature (°C)	B	30	45	60
inhibitor concentration (g/l)	C	0.2	0.4	0.8

Table 2
Matrix of process Variables.

S/N	variable 1 time(h)	variable 2 temp (°C)	variable 3 inh. conc (g/l)
1	8.00	45.00	0.80
2	5.00	45.00	0.60
3	5.00	45.00	0.60
4	3.00	45.00	0.80
5	8.00	60.00	0.60
6	8.00	60.00	0.60
7	5.00	60.00	0.20
8	8.00	45.00	0.80
9	8.00	45.00	0.80
10	3.00	60.00	0.40
11	3.00	60.00	0.40
12	5.00	30.00	0.80
13	3.00	45.00	0.80
14	3.00	60.00	0.40
15	8.00	45.00	0.80
16	8.00	45.00	0.80
17	5.00	30.00	0.20

Table 3
Results of thermometric test.

Inhibitor Concentration (g/L)	RN (°C/min)	IE (%)
1.0	0.02730	
0.2	0.01180	56.77
0.4	0.00711	73.96
0.6	0.00425	84.43
0.8	0.00352	85.11

Table 4
Result of phytochemical analysis.

S/N	Substances	Presence
1	Saponins	++
2	Tannis	+
3	Flavonoids	+
4	Alkaloids	-
5	Phenolics	-
6	Glycosides	++

Where.
= not present.
+ = present.
++ = highly present.

Table 5
Results of weight loss measurement.

S/N	Factor 1 A: time(h)	Factor 2 B: temp (°C)	Factor 3 C: Conc (g/l)	WT Loss	CR (mg/mm ² h)	IE	Sur. coverage
1	7.00	45.00	0.80	0.286	0.970	50.940	0.509
2	5.00	45.00	0.60	0.372	0.178	66.490	0.665
3	5.00	45.00	0.60	0.372	0.178	66.490	0.665
4	3.00	45.00	0.80	0.288	2.290	42.360	0.424
5	7.00	60.00	0.60	0.346	1.200	51.860	0.519
6	7.00	60.00	0.60	0.346	1.200	51.860	0.519
7	5.00	60.00	0.20	0.355	1.600	50.780	0.508
8	7.00	45.00	0.80	0.286	0.970	50.940	0.509
9	7.00	45.00	0.80	0.286	0.970	50.940	0.509
10	5.00	60.00	0.40	0.474	3.770	84.710	0.847
11	3.00	60.00	0.40	0.474	3.770	78.920	0.789
12	5.00	30.00	0.80	0.344	1.640	60.640	0.606
13	3.00	45.00	0.80	0.288	2.290	42.360	0.424
14	3.00	60.00	0.40	0.474	3.770	78.920	0.789
15	7.00	45.00	0.80	0.286	0.970	50.940	0.5090
16	7.00	45.00	0.80	0.286	0.970	50.94	0.5094
17	5.00	30.00	0.20	0.4209	2.010	74.62	0.7462

3.5. Results of the 3D plots

Figs. 2–4 illustrates the 3-D plots for this study.

The inhibition increased with decreasing time as shown in Fig. 2. According to Fig. 3, inhibitor efficiency increased with temperature.

Table 6
ANOVA.

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Model	2269.12	8	283.64	3.62	0.0435	significant
A	4.17	1	4.17	0.053	0.8232	
B	9.60	1	9.60	0.12	0.7352	
C	0.52	1	0.52	6.596	0.9373	
A2	105.92	1	105.92	1.35	0.2782	
B2	421.36	1	421.36	5.39	0.0489	
C2	559.94	1	559.94	7.16	0.0281	
AB	1338.81	1	1338.81	17.11	0.0033	
AC	0.000	0				
BC	35.34	1	35.34	0.45	0.5205	
Pure Error	625.97	8	78.25			
Cor Total	2895.09	16				

R-Squared 0.7838
Adj R-Squared 0.5676

DESIGN-EXPERT Plot
Response 1

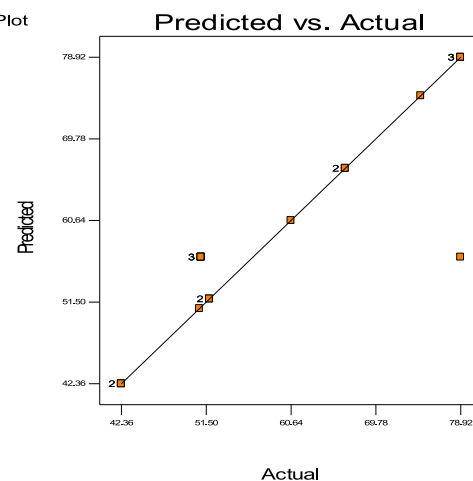


Fig. 1. Graph of predicted versus actual.

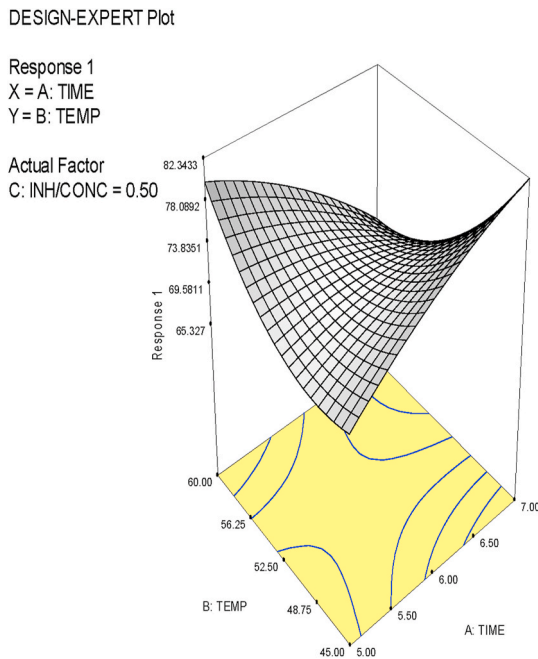


Fig. 2. 3D plot of temp and time.

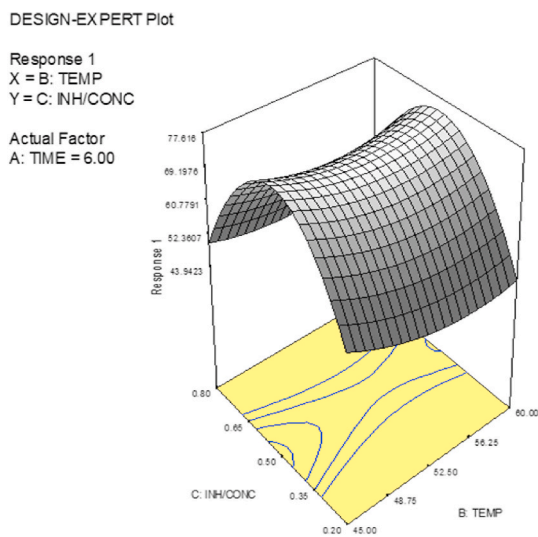


Fig. 3. 3D Plot of Inh conc and Temp.

Fig. 4 shows that increasing the temperature reduced inhibitor efficiency. Metal corrodes more quickly the longer it is exposed to an acidic medium at a higher temperature which explains why [12,34] respondents agreed with the study's findings.

3.6. Validated experiment

The optimal process level was subjected to experimental validation. The Inhibition efficiency observed was 86.42%.

3.7. Results of scanning electron microscope (SEM)

SEM results for blank, coupon with the maximum inhibitory efficiency, and coupon seen at the optimal process level were shown in Figs. 5–7. Pitting corrosion was seen in Fig. 5, and an outer film with fewer cracks, which indicated less corrosion damage was seen in Fig. 6. Additionally, as illustrated in Fig. 7, additional film that served as a

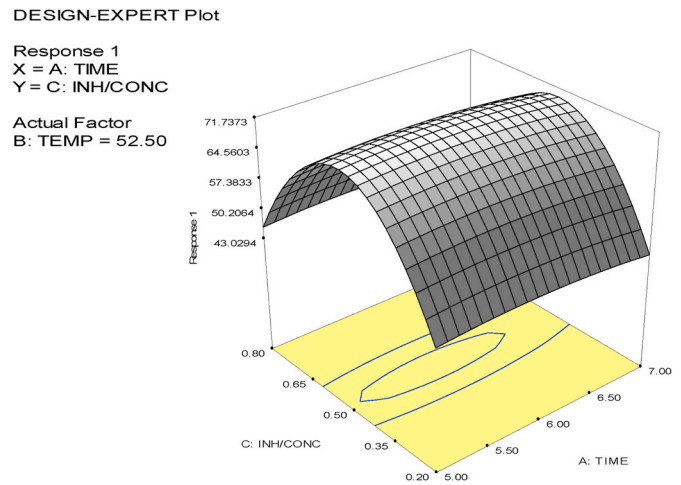


Fig. 4. 3D plot of inhibitor conc. And time.

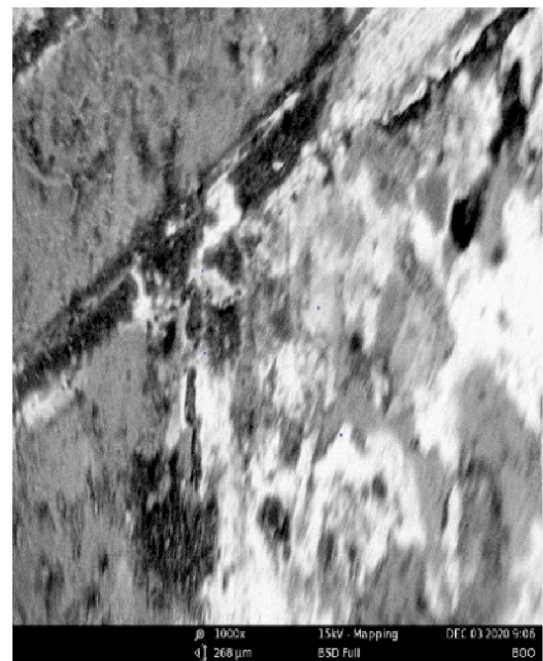


Fig. 5. Blank coupon.

corrosion barrier was generated on the confirmed optimal process level coupon.

3.7.1. Inhibition mechanism

The metal gains a positive charge due to metal dissolution at the anodic area of the RS molecule protonated in H₂SO₄ solution, while H²⁺ ions from the solution are reduced at the cathodic region. The negatively charged SO₄²⁻ ion from H₂SO₄ is drawn to this positively charged metal surface, creating an electrical double layer at the metal solution interface. In contrast to chemical adsorption, which is caused by interactions between donors and acceptors, this double layer promotes the electrostatic interaction-based adsorption of protonated RS molecules onto it.

4. Conclusions

Rice straw extract contained bioactive constituents of a good corrosion inhibitor; Optimal process level observed was: Time: 5.49 h, Temperature: 55 °C; Concentration: 0.47 g/l with Inhibition efficiency of

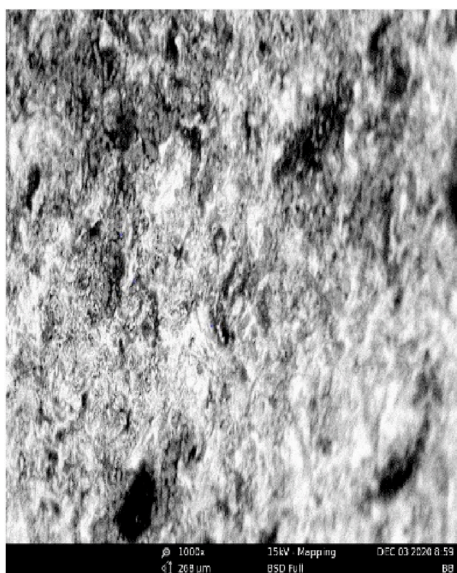


Fig. 6. Coupon with highest IE.



Fig. 7. Coupon from optimal process.

86.42 while 84.71% was the maximum inhibition efficiency observed from the experimental design.

The SEM analysis revealed that more passive film acted as corrosion barrier on surface of coupon from validated experiment. This confirmed that rice straw extract was a good corrosion inhibitor. This research can benefit manufacturing industries which used mild steel as the major material.

The roles of authors

Conceptualization: **Olamide OYEWOLE**. Data curation: **Olamide OYEWOLE**, **T. Siji ABAYOMI**, **Temitope A. OSHIN**. Formal analysis: **Olamide OYEWOLE**, **T. Siji ABAYOMI**, **Toyin A. OREOFE**, **Temitope A. OSHIN**. Investigation and Methodology: **Olamide OYEWOLE**, **T. Siji ABAYOMI**. Project administration: **Olamide OYEWOLE**, **T. Siji ABAYOMI**, **Toyin A. OREOFE**, **Temitope A. OSHIN**. Resources,

Software: **Olamide OYEWOLE**, **T. Siji ABAYOMI**, **Toyin A. OREOFE**, **Temitope A. OSHIN**. Validation: **Olamide OYEWOLE**, **T. Siji ABAYOMI**, **Toyin A. OREOFE**, **Temitope A. OSHIN**. Writing - original draft: **Olamide OYEWOLE**. Writing - review and editing: **Olamide OYEWOLE**, **Temitope A. OSHIN**.

Declaration of competing interest

The authors declare that they have no competing interest.

Data availability

Data will be made available on request.

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