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THE USE OF BANANA STEM EXTRACT AS GREEN INHIBITOR TO MITIGATE CORROSION IN ACIDIC ENVIRONMENT: OPTIMIZATION APPROACH

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ABSTRACT

Organic corrosion inhibitors, which are commonly used in the petroleum, refinery, pipeline, and automobile applications, are toxic and have negative environmental consequences. A cost-effective green inhibitor is undoubtedly a better option. The aim of this study is to use banana stem extract as a viable green inhibitor to mitigate corrosion in marine environment using an optimization approach. The extract was phytochemically analyzed to find out if it contained any bioactive constituents capable of preventing metal corrosion. Box-Behnken design was used to investigate the effects of process variables: temperature (30°-60°C), immersion time (3-9 days), extract amount (0.2-0.8 g/l). Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopy (EDS) were employed for characterization. The extract was found to be a good inhibitor because it contains alkaloids, phenols, tannins, saponins, terpenoids, steroids and flavonoids, according to the results of the phytochemical analysis. The experimental design's optimal process levels were found to be 45°C, 6 days of immersion, and 0.5 g/l of inhibitor. It was observed that more white patches were present on the SEM and EDS results of the mild steel from validated experiment via adsorption. It confirmed that banana stem extract is a good inhibitor in 0.1M H_2SO_4 solution.

Keywords: *Corrosion, Optimization, Phytochemical Analysis, Energy Dispersive Spectroscopy*

1. INTRODUCTION

Green corrosion inhibitors are free of hazardous substances, biodegradable materials, and heavy metals (Alimohammadi et al., 2023), and have been reported to be the best technique for mitigating corrosion challenges. It has been found that the presence of organic compounds such as flavonoids, steroids, tannins and alkaloids make some of these plant-biowastes to have inhibitory effects. Several researchers claim that inhibitor molecules adhere to metal surfaces and stop corrosion in corrosive solutions (Qiang et al., 2023).

Some of the plant extracts that had been used as corrosion inhibitor are: Bitter kola (Anadebe et al., 2018); Polyphenol extract (Chami et al., 2023); maple leaves extract (Wang et al., 2023); waste feverfew root (Zhou et al., 2023); *Pisum sativum L* leaves extract (Chen et al., 2023); *Glebionis coronaria* plant extract (Kellal et al., 2023); *Convolvulus microphyllus* extract (Hladhar et al., 2023); Tulsi and green tea extracts (Chowdhury et al., 2023); *Chromolaena odorata* leaves extract (Liao et al., 2023); Papaya leaves extract (Tan et al., 2021); *Medicago sative* plant (Al-Turkustani et al, 2011); *Moringa oleifera* and *Jatropha curcas* leaves extracts (Ikubnani et al., 2023); essential oil extracted from the leaves (El Aatiaoui et al., 2023); walnut green husk extract (Li et al., 2023*), Stylosanthes gracilis* extract (Ofuyekpone etal., 2021); Methyl-5-benzoyl-2-benzimidazole Carbamate (Mebendazole) (Edoziuno et al., 2020); extract of *Centrosema pubescens* (Ofuyekpone et al., 2023); Anthelmintic Drug (Edoziuno et al., 2024). There are large numbers of

inhibitors reported in corrosion science but no work had been reported on the use of Banana stem extract as a corrosion inhibitor in 0.1 M H_2SO_4 environment with optimization and optimization studies as at when this research was conducted. This study investigated the use of banana stem extract as green inhibitor to mitigate corrosion in acidic environment via optimization approach.

2. MATERIALS AND METHODS

2.1 Preparation of Banana Stem Extract (BSE)

The Landmark University teaching and research farm provided the banana stem which was air-dried for 4 days to remove moisture and retain the bioactive components. 35 grams of the powdered banana stem was weighed and extracted using 350 ml of ethanol as solvent at a temperature of 78 ºC for 4 hours through a Soxhlet extraction process. The entire process was repeated and 500ml of banana extract was gotten which was used for the research. Thereafter, the extract was kept in the refrigerator for further use.

2.2 Preparation of mild steel

Mild steel was cut into 2.2 cm by 1.9 cm coupons with a centrally punched hole measuring 0.1 cm. Emery paper was used to scrub mild steel in order to remove any dirt from the metal surfaces of the samples. Prior to being air-dried and weighed, the samples were washed in distilled water and cleaned with acetone.

2.3 Design of Experiment

Box-Behnken Design was used to study the interactions of process variables. The variables considered were: Inhibitor concentration (0.2 g/l-0.8g /l), Temperature (30 ºC-60 ºC) and Time (3 days-9 days) which generated 17 experimental runs. This methodology was adapted from Oyewole et al. (2022) and Oyewole et al. (2023b). Table 1 shows the variables that were tested and levels. Box-Behnken Design for the interaction of variables for the 17 experimental runs that were generated is in Table 2.

Table 1: Variables with different levels

Variables	Unit	Levels	
		Low	High
Time of immersion	Days	3	9
Temperature	^{0}C	30	60
Inhibitor concentration	g/l	0.2	0.8

Table 2: Box-Behnken Design for interaction of variables

	Variable 1	Variable 2	Variable 3
Run	A: Temperature (°C)	B: Inhibitor Concentration (g/l)	C: Time (days)
1	45	0.5	6
2	45	0.5	6
3	45	0.8	3
4	60	0.5	3
5	45	0.2	3
6	45	0.8	9
7	45	0.2	9
8	45	0.5	6
9	45	0.5	6
10	30	0.5	3
11	60	0.2	6
12	30	0.8	6
13	30	0.5	9
14	60	0.5	9
15	60	0.8	6
16	45	0.5	6
17	30	0.2	6

2.4 Phytochemical Analysis

The phytochemical analysis of the banana stem extract was performed to determine the presence of bioactive constituents.

2.4.1 Test for Tannins

In a water bath containing 30 cm^3 of water, 0.30 g of (Banana Stem Extract) BSE was weighed and boiled for 10 minutes. Filtration was carried out after boiling using Whatman filter paper (125 mm). Three drops of 0.1 % ferric chloride were applied to 5 cm^3 of the filtrate. This methodology was adopted from Ejikeme et al. (2014).

2.4.2 Test for Saponins

Water bath with a volume of 30 cm3 of water, 0.30 g of BSE was boiled for 10 minutes and filtered using Whatman filter paper (125 mm). A mixture of 5 cm3 of distilled water with 10 cm3 of filtrate was centrifuged for a stable persistent froth. This analysis was adopted from Ejikeme et al. (2014).

2.4.3 Test for Terpenoids

To 0.5 ml of BSE, 1 ml of trichloroacetic acid was added. The appearance of red colouration indicated the presence of terpenoids as adopted from (Ahmed et al., 2022; Kancherla et al., 2019).

2.4.4 Test for Flavonoids

BSE (0.30 g) was weighed and transferred into a beaker. The BSE was extracted with 30 cm3 of distilled water at room temperature, for 2 hours and filtered with Whatman filter paper (125 mm). 10 cm3 of the aqueous filtrate of the BSE was added to 5 cm3 of 1.0 M of dilute ammonia solution, thereafter; 5 cm3 of concentrated tetraoxosulphate (VI) acid was added. This method was adopted from Ezeonu and Ejikeme (2016).

2.4.5 Test for Alkaloids
Diluted HCl was added to 2 g of BSE, then mixed evenly and filtered. Mayer reagent was added to 2-3 ml of the filtrate and Mayer's test was conducted. The formation of a yellow precipitate showed Alkaloid was present. This method was adopted from (Parbuntari et al., 2018).

2.4.6 Test for Steroids
2 ml of chloroform was added to 2 ml of BSE and 1 ml of concentrated H_2SO_4 was added by the side of the test tube. After shaking, the formation of red colouration in the upper layer and a greenish-yellow fluorescence in the acid layer indicates the presence of steroids.

2.5 Weight Loss Analysis
The weight loss was calculated using the experimental runs generated by the software.
The weight loss was calculated using equation (1)

$$\Delta W = Wp - Wf \quad (1)$$

where ΔW is the weight loss (g),
Wp is the weight before immersion (g);
Wf is the weight after immersion (g).
The corrosion rate in the absence and presence of inhibitors was calculated using equation 2.;

$$CR = \frac{\Delta W}{At} \quad (2)$$

t is time of exposure in days
A is area of the specimen (cm^2)
CR is the corrosion rate at each exposure time.

2.6 Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) Characterization
SEM-EDS were used to characterize and determine the surface morphology and elemental compositions for mild steel of: optimal process level (validated); blank and maximum inhibition efficiency. The type of SEM-EDS used was JCOL-model JSM-6390 at Covenant University, Ota, Nigeria.

3. RESULTS AND DISCUSSION

3.1 Result of Phytochemical Analysis
Table 3 displayed the findings of phytochemical analyses conducted on banana stem extract. This demonstrated the presence of alkaloids, terpenoids, flavonoids, phenols, tannins, and saponins. These components were reported to be components of a good inhibitor in a number of green inhibitors made from plant biomass, supporting the research result of (Oyewole et al., 2023a).

Table 3: Phytochemical Analysis

S/N	Phytochemical constituents	Result
1	Tannnis	+
2	Saponins	+
3	Steroids	+
4	Terpenoids	+
5	Flavonoids	+
6	Alkaloids	+

+ indicated presence of phytochemicals
- indicated absence of phytochemicals

3.2 Result of Weight Loss Measurements
The tests were performed under total immersion. The results of the third experimental run showed the lowest corrosion rate. Table 4 displays the weight loss analysis results, while Table 5 displays the inhibition efficiency results. The conditions of 45 ºC, 6 days, and 0.5 g/l of inhibitor concentration resulted in highest level of inhibition efficiency.

Table 4: Result from Weight Loss Measurement

		Variable 1	Variable 2	Variable 3	Response 2	Response 3
Std	**Run**	**A: Temperature (°C)**	**B: Inhibitor Concentration (g/l)**	**C: Time (days)**	**Corrosion Rate (g/days.cm²)**	**Weight Loss (grams)**
8	1	60	0.5	9	0.0130	0.4890
3	2	30	0.8	6	0.0061	0.1530
11	3	45	0.2	9	0.0022	0.0840
9	4	45	0.2	3	0.0136	0.1700
10	5	45	0.8	3	0.0200	0.2590
2	6	60	0.2	6	0.0046	0.1160
14	7	45	0.5	6	0.0024	0.0600
7	8	30	0.5	9	0.0037	0.1380
1	9	30	0.2	6	0.0082	0.2060
5	10	30	0.5	3	0.0152	0.1900
6	11	60	0.5	3	0.0093	0.1170
17	12	45	0.5	6	0.0024	0.0600

Std	Run	Variable 1	Variable 2	Variable 3	Response 2	Response 3
		A: Temperature (°C)	**B: Inhibitor Concentration (g/l)**	**C: Time (days)**	**Corrosion Rate (g/days.cm^2)**	**Weight Loss (grams)**
16	13	45	0.5	6	0.0024	0.0600
13	14	45	0.5	6	0.0024	0.0600
12	15	45	0.8	9	0.0041	0.1530
4	16	60	0.8	6	0.0051	0.1270
15	17	45	0.5	6	0.0024	0.0600

Table 5: Result of Inhibition efficiency

Std	Run	Variable 1	Variable 2	Variable 3	Response 1
		A: Temperature (°C)	B: Inhibitor Concentration (g/l)	C: Time (days)	Inhibitor Efficiency
8	1	60	0.5	9	92.9058
3	2	30	0.8	6	97.7638
11	3	45	0.2	9	98.2989
9	4	45	0.2	3	83.0000
10	5	45	0.8	3	96.2590
2	6	60	0.2	6	98.1220
14	7	45	0.5	6	98.5667
7	8	30	0.5	9	98.0069
1	9	30	0.2	6	97.0115
5	10	30	0.5	3	95.6640
6	11	60	0.5	3	98.1798
17	12	45	0.5	6	98.5667
16	13	45	0.5	6	98.5667
13	14	45	0.5	6	98.5667
12	15	45	0.8	9	97.8011
4	16	60	0.8	6	98.2380
15	17	45	0.5	6	98.5667

3.3 Result of ANOVA for the Corrosion Rate, CR

The model is significant, according to the model's F-value of 4.70. The probability that an F-value this large could be the result of noise is only 2.67%. **P-values** less than 0.0500 indicate model terms which are significant. In this case **C^2** and AC are the significant term. The Adjusted R^2 of 0.6757 and coefficient of regression R^2 was 0.8581 were observed from the ANOVA as shown in Table 6. Regression equations generated in terms of coded terms is in equation 3 respectively. The result of ANOVA is in Table 6.

Regression Equation

$$\text{Corrosion rate} = 0.0024 - 0.001A + 0.0008B - 0.0044C - 0.0006AB + 0.0038AC - 0.0012BC + 0.0020A^2 + 0.0016B^2 + 0.0059C^2 \quad (3)$$

Table 6: ANOVA

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	0.0004	9	0.0000	4.70	0.0267	significant
A-temperature	1.554	1	1.554	0.0159	0.9031	
B-inhibitor concentration	5.532	1	5.532	0.5672	0.4759	
C-Time	0.0002	1	0.0002	15.81	0.0054	
AB	1.629	1	1.629	0.1670	0.6950	
AC	0.0001	1	0.0001	5.88	0.0451	
BC	5.428	1	5.428	0.5565	0.4800	
A^2	0.0000	1	0.0000	1.66	0.2390	

B²	0.0000	1	0.0000	1.17	0.3144
C²	0.0001	1	0.0001	15.21	0.0059
Residual	0.0001	7	9.753		
Lack of Fit	0.0001	3	0.0000		
Pure Error	0.0000	4	0.0000		
Cor Total	0.0005				
		Adj R² 0.6757	R^2	0.8581	

3.4 Surface Response Plots

The 3D interactive effect of process variables on corrosion rate are shown in Figures 1–3. These showed relationship between the variables and corrosion rate response.

3.5 Experimental Validation

The design of experiment predicted variables optimal process level as Time: 7.03 days, Temp: 55.3ºC and Conc: 0.62g/l. This has been validated, and an inhibition efficiency of 96.935% was obtained.

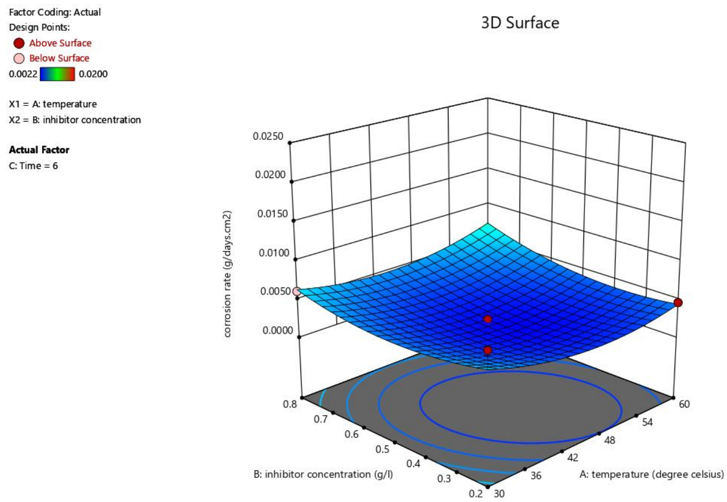


Figure 1: 3-D Response Surface Plot for the interaction of Temperature and Inhibition Conc

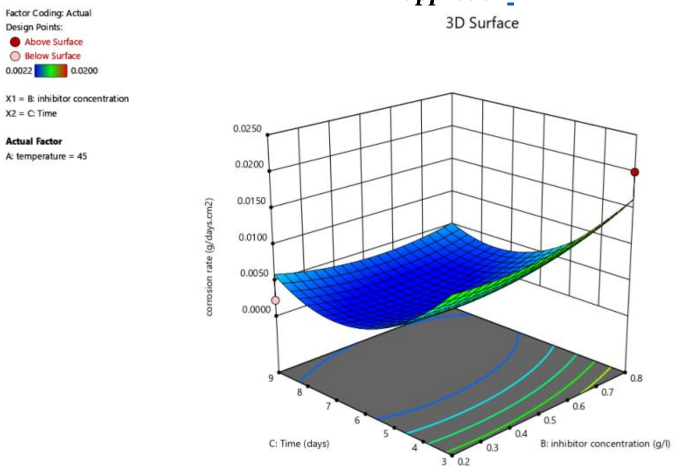


Figure 2: 3-D Response Surface Plot for the interaction of Time and Inhibition Conc

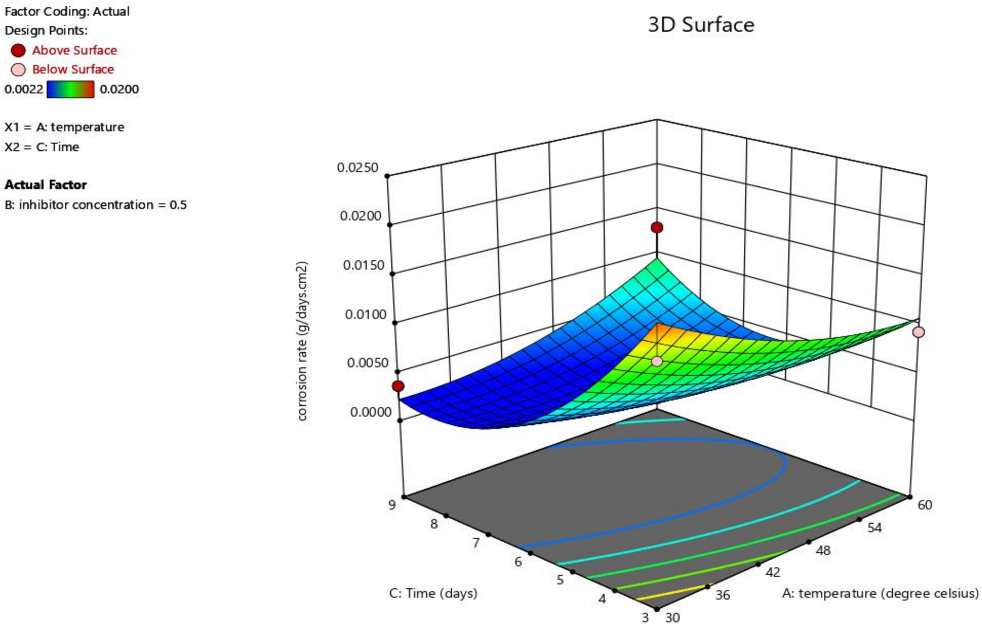
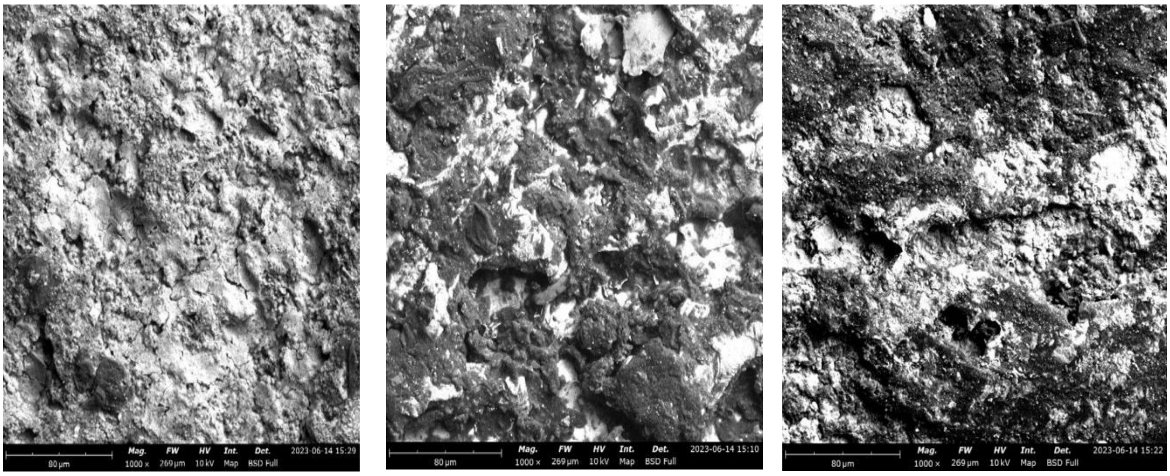


Figure 3: 3-D Response Surface Plot for the interaction of Temperature and Time

3.6 Results of Scanning Electron Microscope (SEM) Analysis

The acidic medium caused the blank surface to be severely damaged, as shown in Figure4a, with significant cracks and serrated edges. In contrast, Figure 4b, showed less damage and smoother surface, due to inhibitor present. In addition, Figure 4c demonstrated a more protective bio-film which was caused by interaction of bioactive constituents on the outer layer of mild steel at the optimal process level (validated).

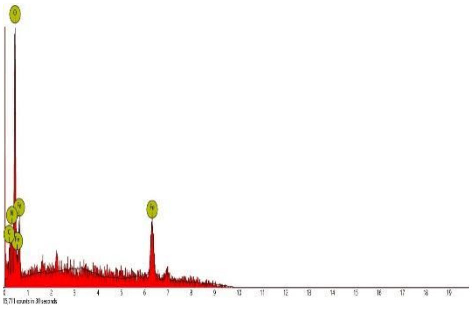


4a 4b 4c

Figure 4a: Blank surface morphology
Figure 4b: Mild steel with highest inhibition efficiency, morphology
Figure 4c: Mild steel of validated experiment surface morphology.

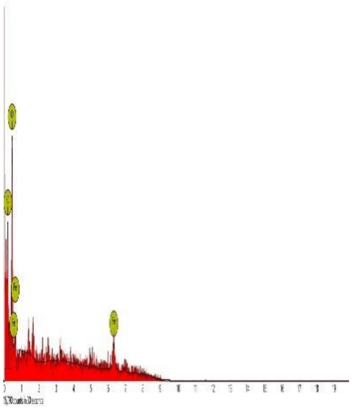
3.7 Results of EDS Analysis

EDS was used to examine the elemental compositions of mild steel blank, mild steel with the highest inhibition efficiency, and mild steel with optimal process variables (validated), as illustrated in Figures 5a-c. The elemental composition of the blank showed presence of O, Fe, N, and C; in Figure 5a. O, Fe, higher amount of O which is a heteroatom was seen in Figure 5b. There is decrease in amount of O while amount of Fe increased as seen in Figure 4c, which supported the findings of (Alimohammadi et al., 2023; Oyewole et al., 2023b, Abaei, et al., 2023). Furthermore, in the validated experiment, interphase functioned as a physical barrier to further impede corrosion in the mild steel. H2 + BSE was the van der Waals interaction, and the extract adsorbed $SO4^{-}$ ion. As a concentrated oxide hydroxide inhibition component, the corrosion and
proactive types' active site was blocked.



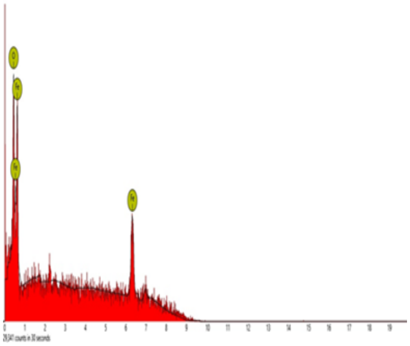
Element symbol	Element name	Atomic conc %	Weig %
O	Oxygen	54.57	29.05
Fe	Iron	35.85	66.61
N	Nitrogen	7.75	3.61
C	Carbon	1.83	0.73

Figure 5a: EDS spectra of the mild steel of the Blan



Element symbol	Element name	Atomic conc %	Weight conc %
O	Oxygen	63.89	39.83
Fe	Iron	25.33	55.12
C	Carbon	10.78	5.05

Figure 5b: EDS spectra of mild steel obtained via highest inhibition efficiency



Element symbol	Element name	Atomic conc %	Weight con[c] %
O	**Oxygen**	**40.21**	**16.15**
Fe	**Iron**	**59.79**	**83.85**

Figure 5c: EDS spectra of mild steel of optimal process level

4. CONCLUSIONS

The presence of saponins, flavonoids, tannins, phenols, steroids, terpenoids, and alkaloids was revealed by the phytochemical analysis, confirming the effectiveness of banana stem extract as a corrosion inhibitor. The validated experiment was carried out at an optimal process level of 7.03 days, 55.33 ºC and 0.62 g/l inhibitor concentration. The result of SEM and EDS showed that more protective film was formed on the surface of the mild steel. This confirmed that more adsorption was responsible for the blockage of corrosion on mild steel of validated experiment than from highest inhibition efficiency. It can be concluded that Banana stem is a good inhibitor for the corrosion of mild steel in 0.1M H_2S0_4. This research can be replicated to solve corrosion problems in petroleum, oil and service industries because it is cost effective and also an eco-friendly approach.

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