



Research article

Corchorus olitorius stem as corrosion inhibitor on mild steel in sulphuric acidO. Oyewole^{a,b,*}, T.A. Oshin^a, B.O. Atotuoma^a^a Department of Chemical Engineering, Landmark University, PMB 1001 Omu-Aran, Kwara State, Nigeria^b SGD 9:Industry, Innovation and Infrastructure Research Group, USA

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ABSTRACT

The corrosion inhibition of mild steel in 0.5M H₂SO₄ solution was studied in the presence of *Corchorus olitorius* stem extract as inhibitor. Phytochemical analysis results confirmed the presence of organic constituents such as Alkaloids, Tannins, Glycosides, Saponins and Flavonoids which made the *Corchorus olitorius* stem extract act as a good inhibitor. The highest inhibition efficiency as observed from the experimental design was 93.29%, with process levels of 4 days, temperature: 60 °C and inhibitor concentration: 1.0 g/L respectively. The optimal process levels were: 4.11 days, temperature: 48.92 °C and inhibitor concentration: 1.16 g/L respectively, which gave an inhibition efficiency of 94.34%. The result from the Scanning Electron Micrographs showed that via the validated experiment, a more passive layer of film was formed on the surface of the coupon, confirming the high efficiency of the *Corchorus olitorius* extract's inhibiting role in corrosion prevention.

1. Introduction

The petroleum industry is one of the most impacted by corrosion, which affects machinery and pipelines from crude oil production to final product transportation, due to the presence of various corrosive substances in crude oil (Obot et al., 2009). Furthermore, because corrosion is a diffusion-controlled process, it occurs on exposed surfaces. As a result, different methods to combat corrosion are being explored, the major one being green corrosion inhibitors due to their eco-friendly reaction with the environment. Since inhibitors form films in many ways, such as by adsorption and the formation of a passive layer on the metal surface, they have been extensively studied for reducing corrosion of mild steel in acidic environments. Moreover, in a corrosive setting, a corrosion inhibitor is a material that effectively inhibits or prevents the corrosion of exposed metal (Popov, 2015). Researches had been tailored to the use of agricultural wastes which are also known as green inhibitors. This is because they are environmentally friendly and readily available. Some of the green inhibitors are: extract of *C. papaya* leaves (Kavitha et al., 2014); extract of *Hyptis suaveolens* leaves (Muthukrishnan et al., 2014); *Xanthium strumarium* leaves extract (Khadom et al., 2018); Juniperus plants (Al-Mhyawi, 2014); Pawpaw leaves (Omotioma and Onukwuli, 2017); Bamboo leaves (Li et al., 2012, 2014); Mango extract (Onukwuli and Monday, 2016); *Origanum majorana* Extracts (Challouf et al., 2016); Pigeon pea leaf (Anadebe et al., 2019); *Nypa fruticans wurnb* leaves extract (Michael and Olubunmi, 2014); *Aniba rosacodora* plant extract (Chevalier

et al., 2014); *Phyllanthus fraternus* leaves extract (Patel et al., 2014); *Gentiana olivieri* extract (Baran et al., 2016); *Polyalthia longifolia* leaves (Vasudha and Priya, 2014); *Eucalyptus* leaf extract (Abdel-Gaber et al., 2020); Extract of *Olea europaea* L. Leaves (Düdükcü et al., 2020); extract of *Salvia officinalis* leaves (Soltani et al., 2012); *Osmanthus fragran* leaves extract (Li et al., 2012); an apple-based green (Mehdi et al., 2020); coconut leaves (Siyi et al., 2020); *Commiphora caudata* (Mohanraj and Aejitha, 2020); Watermelon waste (Odewunmi et al., 2015); *Talinum triangulare* (Vincent and Chinedu 2020); *Neem* leaves extract (Tuaweri et al., 2015); *Sida acuta* (Umoren et al., 2016); *Aloe Barbadensis* (*Aloe vera*) extract (Pankaj and Gargi, 2014); *Ficus exasperata* extract (Oyewole et al., 2021).

The serious consequences of corrosion have become an issue of worldwide significance. In recent years, the use and development of various sustainable inhibitors has become a subject for many researchers. The current goal is to develop eco-friendly, low-cost corrosion inhibitors made from plant extracts as a ready-to-use renewable resource. Several researchers have looked into the effectiveness of green corrosion inhibitors derived from plants for metal corrosion control in acidic environments. The optimization method of the use of *Corchorus olitorius* stem extract in 0.5 M sulphuric acid has not previously been exploited as a corrosion inhibitor. The output of the *Corchorus olitorius* stem extract in 0.5 M sulphuric acid using optimization strategy and measurements of weight loss is therefore important and essential to research. This extract was used because it is green and economically efficient. This research

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Table 1. Experimental ranges in coded terms and actual value ranges.

Independent variables	Codes	Low value	High value
Time (days)	X ₁	2	6
Temperature (°C)	X ₂	30	60
Inhibition Concentration (g/L)	X ₃	0.5	1.5

investigates the use of *Corchorus olitorius* stem as a corrosion inhibitor on mild steel in 0.5M H₂SO₄ solution.

2. Materials and methods

2.1. Preparation of specimen

The mild steel used in this experiment was collected from Landmark University's mechanical workshop in Omu-Aran, Kwara State, Nigeria. The mild steel was cut into coupons with dimensions of 1.9cm by 2.2cm, a thickness of 2.2cm, and a 1.5cm hole drilled in the center. After cleaning with emery paper to expose the gleaming surface, the coupons were washed with distilled water, degreased with acetone to remove any oil impurities, and put in a desiccator.

2.2. Preparation of the extract

Fresh *Corchorus olitorius* stem was collected from the cafeteria at Landmark University, Omu-Aran, Kwara state. The stem was then dried under the sun for some days to remove moisture, thereafter grinded to fine powder and then filtered to obtain very smooth particles of the grinded stem. The fine powder obtained was completely soaked in ethanol solution for about 24 h with intermittent stirring to have a homogeneous solution and the extract was collected through filtering in form of paste. The filtrate was then subjected to a process of evaporation for the removal of excess alcohol. The filtrate obtained was used as inhibitor in pure form. The extract prepared was used for concentration ranges of 0.5–1.5 g/L of 0.5 M H₂SO₄ for test solutions.

Table 2. Design matrix for the variable interactions in coded terms.

Std	Run	Block	Factor 1 A:Time (Days)	Factor 2 B: Temperature (°C)	Factor 3 C:Inhibition concentration (g/L)
17	1	{ 1 }	0	0	0
9	2	{ 1 }	-1	-1	-1
6	3	{ 1 }	-1	-1	-1
15	4	{ 1 }	-1	-1	-1
13	5	{ 1 }	-1	-1	-1
20	6	{ 1 }	-1	-1	-1
7	7	{ 1 }	0	0	0
14	8	{ 1 }	0	0	0
2	9	{ 1 }	0	1	0
16	10	{ 1 }	0	0	0
18	11	{ 1 }	0	0	0
12	12	{ 1 }	-1	-1	1
10	13	{ 1 }	0	1	0
8	14	{ 1 }	0	1	0
11	15	{ 1 }	-1	1	-1
3	16	{ 1 }	-1	-1	-1
5	17	{ 1 }	-1	-1	1
19	18	{ 1 }	1	-1	1
1	19	{ 1 }	0	0	-1.8
4	20	{ 1 }	0	0	-1.8

2.3. Phytochemical analysis

Phytochemical analysis was performed on the powdered *Corchorus olitorius* stem to screen for secondary metabolites such as, Saponins, Flavonoids, Tannins, Glycosides, Alkaloids, and Oxalates.

2.4. Corrosive medium

The sulphuric acid solution was made with distilled water. By diluting sulphuric acid in distilled water, the corrosive media (sulphuric acid) was formed. The extract was used in concentrations ranging from 0.5 to 1.5 g/L.

2.5. Experimental design

2.5.1. Response surface methodology (RSM)

RSM (response surface methodology) is a mathematical technique for examining the interaction of the variables with the response. The variables used in this study were: temperature, time and inhibition concentration respectively. The Design Expert Software was used to analyze the variables.

2.5.2. Experimental design

The Central Composite Design generated 20 experimental runs with the three variables which are: temperature, time of immersion and inhibition concentration respectively. Table 1 shows the experimental ranges in coded terms and the real value ranges. Table 2 shows the matrix relationship for the variables in coded terms. Table 3 shows the matrix interaction of the variables via actual factors.

In this analysis, the following procedures were considered for RSM:

- i. For this research, the Central Composite Design (CCD) was used to generate experimental runs;
- ii. Observing the best process level from the experimental run generated by the software via the response with the highest inhibition efficiency;
- iii. Generating a regression equation expressing the relationship in terms of the actual values and the response;

Table 3. Interactions of the three factors.

Std	Run	Block	Factor 1 A: Time (Days)	Factor 2 B:Temperature (°C)	Factor 3 C:Inhibition concentration (g/l)
17	1	Block 1	4	45	1
9	2	Block 1	2	30	0.5
6	3	Block 1	2	30	0.5
15	4	Block 1	2	30	0.5
13	5	Block 1	2	30	0.5
20	6	Block 1	2	30	0.5
7	7	Block 1	4	45	1
14	8	Block 1	4	45	1
2	9	Block 1	4	60	1
16	10	Block 1	4	45	1
18	11	Block 1	4	45	1
12	12	Block 1	2	30	1.5
10	13	Block 1	4	60	1
8	14	Block 1	4	60	1
11	15	Block 1	2	60	0.5
3	16	Block 1	2	30	0.5
5	17	Block 1	2	30	1.5
19	18	Block 1	6	30	1.5
1	19	Block 1	4	45	0.1
4	20	Block 1	4	45	0.1

Table 4. Phytochemical analysis (qualitative analysis).

S/N	Phytochemicals	Reagent	Color change	Result
1	Alkaloids	Wagner	Brown ppt.	Present
2	Tannins	Ferric chloride (FeCl ₃)	Greenish ppt.	Present
3	Saponins	Distilled water	Persistent foam	Present
4	Flavonoids	Sodium hydroxide (NaOH)	Yellow ppt.	Present
5	Glycosides	Sulphuric acid (H ₂ SO ₄)	Reddish brown ppt	Present
6	Phenol	Iron III chloride (FeCl ₃)	No colour change	Absent

DESIGN-EXPERT Plot

Inhibito efficiency
 X = A: TIME
 Y = B: TEMPERATURE

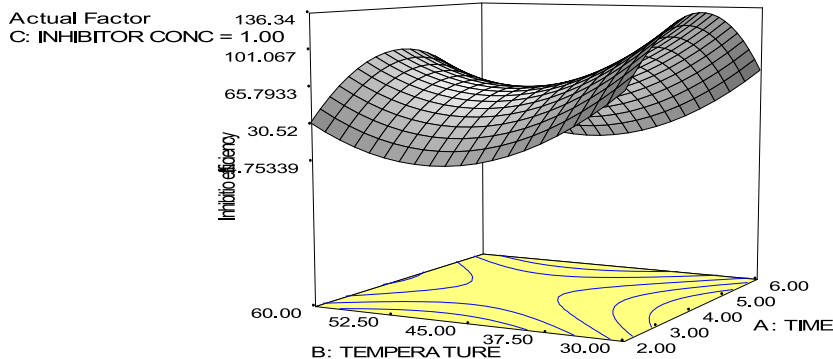


Figure 1. IE versus temperature and time.

DESIGN-EXPERT Plot

Inhibito efficiency
 X = A: TIME
 Y = C: INHIBITOR CONC

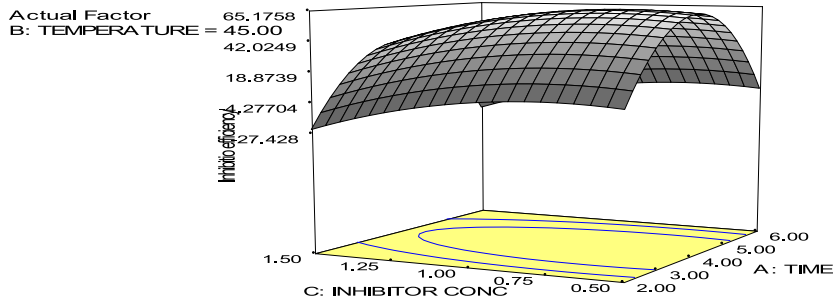


Figure 2. IE versus Inhibition concentration and time.

DESIGN-EXPERT Plot

Inhibito efficiency
 X = B: TEMPERATURE
 Y = C: INHIBITOR CONC

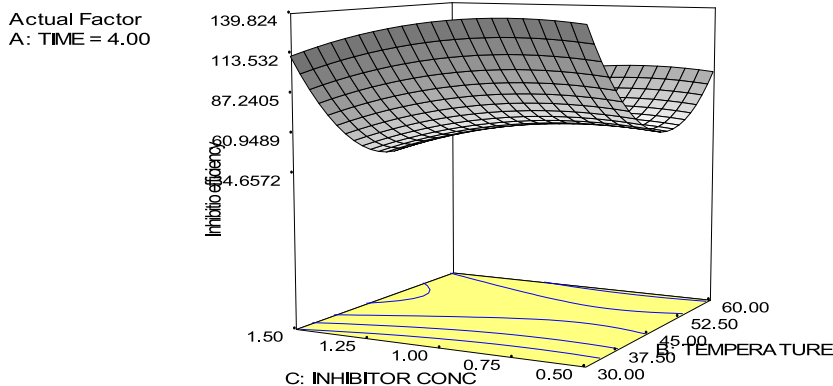


Figure 3. IE versus Inhibition concentration and temperature.

Table 5. The RSM result for the weight loss and corrosion inhibition efficiency of Mild Steel in H_2SO_4 by *Corchorus olitorius* extract.

Run	Temp (°C)	Time (days)	Inhibitor Concentration (g/L)	Weight loss	Inhibition Efficiency (%)	Surface coverage
1	45.00	4.00	1.00	0.0313	61.22	0.6122
2	30.00	2.00	0.50	0.0396	76.03	0.7603
3	30.00	2.00	0.50	0.0396	76.03	0.7603
4	30.00	2.00	0.50	0.0396	76.03	0.7603
5	30.00	2.00	0.50	0.0396	76.03	0.7603
6	30.00	2.00	0.50	0.0396	76.03	0.7603
7	45.00	4.00	1.00	0.0313	61.22	0.6122
8	45.00	4.00	1.00	0.0313	61.22	0.6122
9	60.00	4.00	1.00	0.0511	93.29	0.9329
10	45.00	4.00	1.00	0.0319	62.40	0.6240
11	45.00	4.00	1.00	0.0319	62.40	0.6240
12	30.00	2.00	1.50	0.0270	50.17	0.5017
13	60.00	4.00	1.00	0.0511	93.29	0.9329
14	60.00	4.00	1.00	0.0511	93.29	0.9329
15	60.00	2.00	0.50	0.1728	32.98	0.3298
16	30.00	2.00	0.50	0.0396	76.03	0.7603
17	30.00	2.00	1.50	0.0270	50.17	0.5017
18	30.00	6.00	1.50	0.0242	47.22	0.4722
19	45.00	4.00	1.00	0.0417	80.29	0.8029
20	45.00	4.00	1.00	0.0634	12.37	0.1237

- iv. Prediction of the optimal process levels by the optimization approach in the CCD; and lastly
- v. Experimental validation of the optimal process levels predicted

2.6. Experiment (weight loss analysis)

Weight loss measurements were taken under complete immersion in a thermostatic water bath with a beaker containing a formulated solution at specific temperature ranges.

The coupons were weighed both before and after immersion using time variations, temperature, and inhibitor concentrations for each run as generated by the software. Weight loss (ΔW) was calculated using Eq. (1)

$$\Delta W = W_b - W_a \quad (1)$$

Where W_b is the weight before immersion and W_a is the weight after immersion.

Table 6. ANOVA for the Inhibition efficiency of mild steel in H_2SO_4 by *Corchorus Olitorius*.

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F
Model	5648.37	6	941.40	5.30	0.0057 significant
A	5.80	1	5.80	0.033	0.8593
B	1588.55	1	1588.55	8.95	0.0104
C	1003.11	1	1003.11	5.65	0.0335
A ²	2323.53	1	2323.53	13.09	0.0031
B ²	3420.67	1	3420.67	19.27	0.0007
C ²	1203.82	1	1203.82	6.78	0.0218
AB	0.000	0			
AC	0.000	0			
BC	0.000	0			
Pure Error	2308.23	13	177.56		
Cor Total	7956.61	19			
Std. Dev.	13.33		R-Squared		0.7099
Mean	65.89		Adj R-Squared		0.5760
C.V.	20.22		Pred R-Squared		N/A
PRESS	N/A		Adeq Precision		7.650

While (CR) corrosion rate (g/cm^2h) and Inhibition Efficiency (IE) were calculated using Eqs. (2) and (3) respectively. Where (ΔW) is weight of the coupon in absence and presence of inhibitor, t is the time of immersion and A is the area of the coupon

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

The inhibition efficiency (IE) was calculated using Eq. (3).

$$IE\% = \frac{W_b - W_a}{W_b} \times 100 \quad (3)$$

2.7. Surface analysis

A Scanning Electron Microscope (SEM) was used to study the coupon's surface morphology. The SEM was used to examine the following: the blank coupon, the coupon with the highest inhibition efficiency as determined by the experimental design, and the coupon via the validated optimal process levels.

3. Results and discussion

3.1. Phytochemical analysis of *Corchorus olitorius* extract

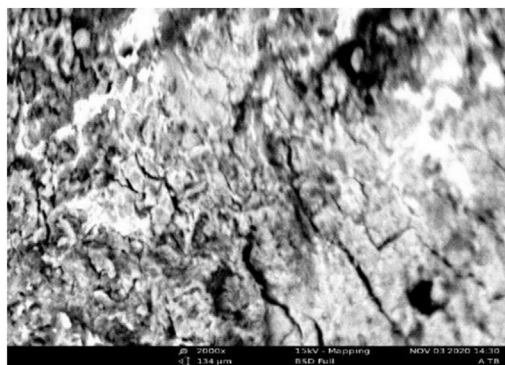
The phytochemical result is as shown in Table 4. It was observed that alkaloids, tannins, saponins, flavonoids and glycosides were present in the extract while phenol was absent. This suggested that extract is a good inhibitor which confirmed the report of Oyewole et al. (2021); Uwah et al. (2013a,b,c)

3.2. 3D surface response plots of the inhibition efficiency using RSM

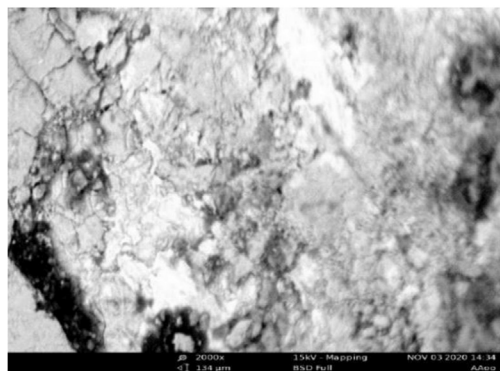
Plotting a three-dimensional surface curve (3D) against any two independent variables, as shown in Figures 1, 2, and 3, was used to investigate the interrelationship between the variables (temperature, time of immersion, and inhibition concentration) and the response (IE).

Inhibition efficiency increases with decreasing time and decreases with increasing temperature, as shown in Figure 1.

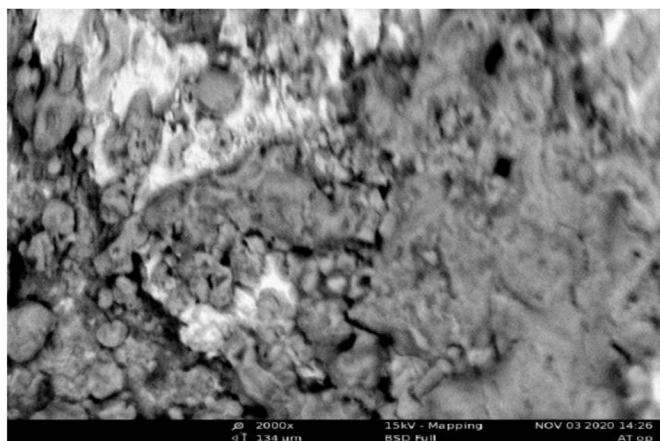
Inhibition efficiency increased with the inhibitor concentration but decreased with the time, as shown in Figure 2, implying that the higher



(a)



(b)



(c)

Figure 4. SEM of mild steel (a) in the absence of inhibitor (blank) (b) at the highest inhibition efficiency and (c) of the validated optimal predicted level.

the inhibitor concentration, the higher the inhibitor efficiency. Figure 3 showed that inhibition efficiency decreased as temperature increases, which can be explained by the fact that the longer the metal is exposed to the acid medium and the higher the temperature, the more it corrodes. The observations from this study confirmed what was reported by Chaitra et al., 2018; Emembolu et al., 2020, Olawale et al., 2019.

3.3. Result of the weight loss measurement using RSM

Table 5 displays the RSM results for weight loss and inhibition efficiency of Mild Steel in H_2SO_4 by *Corchorus olitorius* Extract. The type of design used was Central Composite Design. This design was used to generate the experimental runs via the interactions of the variables considered in this research. In Table 5, the entries 1, 7, and 8 are identical. This is because the difference in the result was too small and when it was approximated to 4 decimal places, the difference did not reflect. The actual weight loss values for Run 1, 7 and 8 are 0.03134, 0.03133 and 0.03139 respectively. The weight loss data of the coupon in acidic environment without the inhibitor (blank coupon) was 0.02236 at 4 days and temperature: 60 °C. This was done because the highest Inhibition Efficiency was at 4 days, temperature: 60 °C.

3.4. Result of the inhibition efficiency IE (%) and statistical analysis as determined using RSM

The Design-Expert software recommended a model based on a higher-order polynomial with significant additional terms. The analysis of variance (ANOVA) for the inhibition of *Corchorus olitorius* extract was shown in Table 6. Model terms are important if the "Prob > F" value is less than 0.0500. B, C, A^2 , B^2 , and C^2 are significant terms.

The significance of the model is indicated by the F value. Temperature had the highest F-value, followed by time, and inhibitor concentration, according to as shown in the ANOVA Table. The coefficient of determination (R^2) of 0.7099 is in agreement with the Adj. R^2 of 0.5760, since the difference is less than 0.2. Moreover, "Adeq Precision" of 7.650 observed implied adequate signal because it is greater than 4. The ANOVA model can be assumed to be statistically valid, and Eq. (7) is the second order polynomial model (regression) equation in terms of the coded values factors of the variables.

Final Equation in Terms of Coded terms

$$\text{Inh Eff} = +61.69 - 1.47A - 21.52B - 12.93C - 62.79A^2 + 53.12B^2 - 11.92C^2 \quad (7)$$

3.5. Scanning electron microscopy

The micrographs of the coupons immersed in H_2SO_4 in the presence and absence of the *Corchorus Olitorius* is shown in Figure 4(a)–(c). It was observed from Figure 4(a) that the blank coupon was highly corroded with cracks, further more, it was observed from Figure 4(b) (best level) gotten from experimental design that there were formation of plain layer with protective film which was as a result of the protective layer formed by the extract, while in Figure 4(c) optimal level that was validated showed more protective film was formed, which can be attributed to adsorption. This result was supported by (Olawale et al., 2019) findings on the inhibiting effects of Chicken Nails Extract (CNE) on mild steel corrosion in 2M H_2SO_4 , as well as Anadebe et al. (2018) findings on mild steel corrosion in acid medium with bitter kola leaf extract as an inhibitor and Anadebe et al. (2019) findings on the application of pigeon pea leaf (PPL) extract as an anti-corrosion agent for mild steel in acid environment (HCl solution).

4. Conclusion

The phytochemical analysis revealed the existence of organic constituents such as Alkaloids, Tannins, Glycosides, Saponins, and Flavonoids, making the *Corchorus olitorius* extract an effective inhibitor.

The impacts of the results are:

> With process levels of 4 days, temperature of 60 °C, and inhibitor concentration of 1.0 g/l, the highest inhibition efficiency was observed in the experimental design, which was 93.29 percent. The

optimal process levels were 4.11 days, 48.92 °C, and 1.16 g/l inhibitor concentration, resulting in a 94.34 percent inhibition efficiency from the validated experiment

> Scanning Electron Micrographs revealed that more passive layer of film was formed on the surface of the coupon via the validated experiment, confirming the high efficiency of the *Corchorus olitorius* extract's inhibiting role in preventing corrosion.

> The results obtained demonstrated the extract's excellent corrosion inhibition properties as a cheap, long-lasting, and cost-effective mild steel inhibitor in sulphuric acid.

> *Corchorus olitorius* extract was found to be an effective inhibitor of mild steel corrosion in 0.5M sulphuric acid.

> The extract acted as a good inhibitor and effective anti-corrosion agent for mild steel in 0.5 M sulphuric acid, which bodes well for future studies.

> The rate of corrosion was found to be reduced as inhibition efficiency improved.

> The extract acted as a strong inhibitor and effective anti-corrosion material for mild steel in 0.5 M sulphuric acid, which augurs well for future study.

> It was discovered that increasing the inhibition efficiency reduced the rate of corrosion.

> This research, however, can be replicated and used to solve corrosion problems in industrial applications due to its cost effectiveness.

Declarations

Author contribution statement

O. Oyewole: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

T. A. Oshin: Performed the experiments; Analyzed and interpreted the data; Wrote the paper.

B. O. Atotuoma: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

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Data availability statement

Data included in article/supplementary material/referenced in article.

Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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