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Optimization and corrosion inhibition of Palm kernel leaves on mild steel in oil and gas applications [☆]

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ABSTRACT

This study investigated the effectiveness of palm kernel leaves extract (PKLE) as green inhibitor using Central Composite Design (CCD). Phytochemical analysis was performed on the extract. Process variables used for the optimization in this study were: concentration of extract (0.5–1.5 g per litre), time (3–5 days), and temperature (30–50 °C) respectively. Surface characterization was done with Scanning Electron Microscope (SEM). Bioactive constituents were observed from the result of the phytochemical analysis. The best process levels were: inhibitor concentration (1.500 g/l), temperature (30 °C) and time (3 days) with inhibition efficiency of 96.74 %; while the optimal process level validated gave 97.20 %. The SEM results revealed that more film was observed on the validated optimal process level. The PKLE extract was an effective inhibitor.

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1. Introduction

Corrosion is a common challenge observed in pipelines in the oil and gas industries which is difficult to eliminate completely. Hence its prevention is a preferred solution that is often sought after [1]. Corrosion inhibition is of utmost necessity to the oil and gas industries since millions of dollars are lost globally due to corrosion and its related activities. The use of inhibitors is promising, inexpensive and is the most effective way of combating corrosion. Inhibitors protect metals by effectively blocking the active sites from being corroded in an environment [2].

Many researches had used different plant extracts as inhibitors because they contain bioactive constituents such as terpenes, alkaloids, flavonoids and phenolic compounds which made them good inhibitors. Some of the extracts reported are: Barely grass extract [3], Bassia muricata extract [4], Petroselinum sativum [5], Citrus limon peel [6], Katemfe leaves [7], Borage flowers [8], Citrus aurantium leaves [9], Ziziphora leaves [10], Pistachio nut [11], Polygonatum odoratum extract [12], Communis oil [13]; Ficus exasperata extract [14]; Vicia faba peel extracts [15] and medicinal materials [16]. This research investigates the corrosive resistance of Palm kernel leaf extracts on mild steel in 0.5 M HCl.

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2. Experimental

2.1. Preparation of the surfaces of the metals

The mild steel used in this experiment was obtained from the University's mechanical workshop. 2 cm × 2 cm × 0.2 cm with 1.5 cm hole drilled in the middle was the dimension of the mild steel. The coupons were cleaned with emery paper, degreased in acetone, and cleaned with a brush and water. After that, it was re-dipped in acetone before air drying.

2.2. Preparation of Palm kernel leaves extract (PKLE)

PKLE was ground into fine powder, after it was dried. To achieve a homogenous solution, the powder was thoroughly soaked in ethanol solution for 24 h and the extract was later collected. Evaporation was used to get rid of any remaining alcohol in the filtrate. The filtrate was employed as an inhibitor in its purest form.

2.3. Phytochemical analysis

Phytochemical study of the PKLE was performed to check for bioactive substances such as alkaloids, flavonoids, terpenoids, tannin, and saponin.

Table 1
Variables with ranges and levels.

Variable	Symbols	Range and levels		
		−1	0	+1
Time (days)	X1	3	4	5
Temperature (°C)	X2	30	40	50
Inhibitor concentration (g/L)	X3	0.5	1.0	1.5

Table 2
Variables' interaction.

Std	Run	A: Inhibitor conc (g/l)	B: Temperature (°C)	C: Exposure Time (days)
6	1	1.50	30.00	5.00
14	2	1.00	40.00	5.00
1	3	0.50	30.00	3.00
10	4	1.50	40.00	4.00
9	5	0.50	40.00	4.00
13	6	1.00	40.00	3.00
5	7	0.50	30.00	5.00
3	8	0.50	50.00	3.00
15	9	1.00	40.00	4.00
20	10	1.00	40.00	4.00
19	11	1.00	40.00	4.00
4	12	1.50	50.00	3.00
16	13	1.00	40.00	4.00
11	14	1.00	30.00	4.00
2	15	1.50	30.00	3.00
18	16	1.00	40.00	4.00
7	17	0.50	50.00	5.00
12	18	1.00	50.00	4.00
8	19	1.50	50.00	5.00
17	20	1.00	40.00	4.00

Table 3
Result of the Phytochemical Analysis of Palm kernel leaves extract.

Bioactive constituents	Qualitative Result	Quantitative Result
Alkaloid	+++	22 mg/g
Saponins	+	0.372 mg/g
Terpenoids	++	16 mg/g
Flavonoids	+++	X
Tannis	+++	X

Where:

X = Not Determined,
 + = Present,
 ++ = Moderately Present,
 +++ = Highly Present.

Table 4
Results of Responses (Inhibition efficiency and corrosion rate).

Std	Run	A: Inhibitor conc. (g/l)	C: Exposure Time (days)	Inhibition Efficiency (%)	Corrosion Rate (g/cm ² h)
6	1	1.50	5.00	93.48	0.0165
14	2	1.00	5.00	75.36	0.017
1	3	0.50	3.00	92.68	0.0075
10	4	1.50	4.00	81.55	0.0119
9	5	0.50	4.00	63.11	0.0238
13	6	1.00	3.00	85.37	0.015
5	7	0.50	5.00	88.41	0.0215
3	8	0.50	3.00	73.17	0.0275
15	9	1.00	4.00	74.75	0.0163
20	10	1.00	4.00	74.75	0.0163
19	11	1.00	4.00	74.75	0.0163
4	12	1.50	3.00	86.16	0.0142
16	13	1.00	4.00	74.75	0.0163
11	14	1.00	4.00	72.82	0.0175
2	15	1.50	3.00	96.74	0.0033
18	16	1.00	4.00	74.75	0.0163
7	17	0.50	5.00	51.45	0.0335
12	18	1.00	4.00	64.08	0.0231
8	19	1.50	5.00	89.86	0.0285
17	20	1.00	4.00	74.75	0.0163

2.4. Weight loss measurements

Weight loss measurement was done with test solutions in thermostatic water bath using matrix interactions generated. Before and after immersion, the weights of the coupons were measured.

Eq. (1) was used to determine weight loss, while equations (2) & (3) are for corrosion rate (CR) (g/cm² days) and Inhibition Efficiency (IE), respectively.

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

The weight before immersion is W_b , while weight after immersion is W_a .

Where (W) is coupon's weight in the absence and presence of the inhibitor, and t is the time

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

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

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

2.5. Experimental design

The Design of Experiment Software 12 version was used to study the process variables interactions. The number of experimental runs generated by the CCD was 20. However, the process variables used in this study were time (3–5 days), extract concentration (0.5–1.5 g/l), and temperature (30–50 °C), with the response being inhibition efficiency (percent) generated. The method was adopted from [14].

Table 1 shows variables with ranges and levels while Table 2 showed variables interactions.

3. Results and Discussion**3.1. Result of Qualitative and quantitative analysis**

Qualitative and quantitative result is shown in Table 3. The presence of alkaloid, saponins, terpenoids, flavonoids, and tannins demonstrated that Palm kernel leaves are a good corrosion inhibitor, confirming a previous report [17].

Table 5
Result of ANOVA.

Sources	Sum of Squares	Df	Mean Square	F Value	p-value Prob > f
Model	2246.84	9	249.65	13.68	0.0002 significant
A-inhibitor conc	623.63	1	623.63	34.17	0.0002
B-temperature	630.59	1	630.59	34.55	0.0002
C-exposure time	126.45	1	126.45	6.93	0.0251
AB	223.34	1	223.34	12.24	0.0057
AC	87.32	1	87.32	4.78	0.0536
BC	13.76	1	13.76	0.75	0.4057
A ²	11.79	1	11.79	0.65	0.4403
B ²	9.00	1	9.00	0.49	0.4985
C ²	280.83	1	280.83	15.39	0.0029
Residual	182.53	10	18.25		
Lack of Fit	182.53	5	36.51		
Pure Error	0.000	5	0.000		
Cor Total	2429.36	19			

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inhibition efficiency

96.74

51.45

X1 = A: inhibitor conc

X2 = B: temperature

Actual Factor

C: exposure time = 4.00

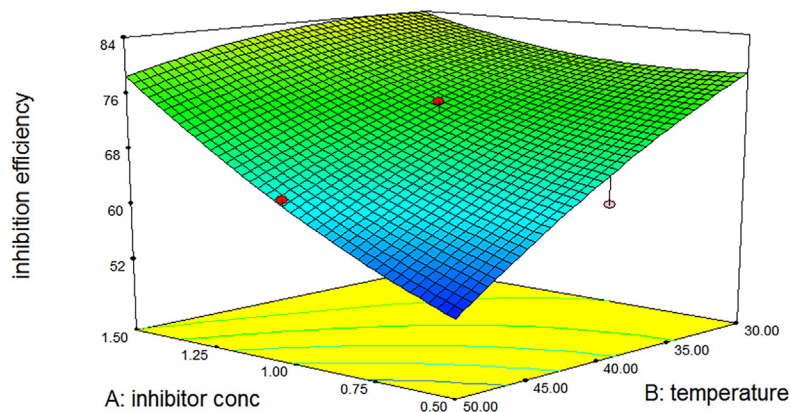


Fig. 1. 3D of Inh..Conc vs Temp.

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inhibition efficiency

96.74

51.45

X1 = A: inhibitor conc

X2 = C: exposure time

Actual Factor

B: temperature = 40.00

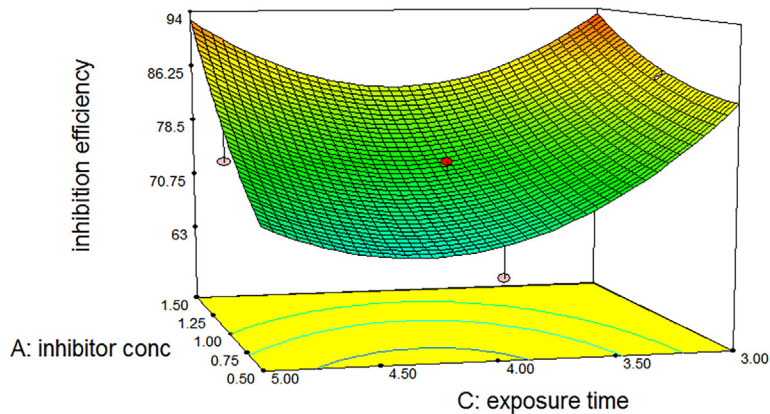


Fig. 2. 3D of inhibitor conc. vsTime.

3.2. Result of experimental design

The result of Responses (Inhibition efficiency and corrosion rate) generated by Experimental Design are presented in Table 4.

3.3. Regression model for the inhibition efficiency (%)

The interactions are designed to maximize the concentration, temperature and time conditions of the system. A well-defined second-order general model is represented by Eq. (4)

$$\hat{y} = \hat{\beta}_0 + \sum_{i=1}^k \hat{\beta}_i x_i + \sum_{i=1}^k \hat{\beta}_{ii} x_i^2 + \sum_{i=1}^k \sum_{j=i+1}^k \hat{\beta}_{ij} x_i x_j \quad (4)$$

Where x_i and x_j (development variables) and β are the tuning parameters represented. The coefficient of determination (R^2)

was 0.9249 in Table 5. The regression equations are in Equations (5) and (6) respectively

$$\begin{aligned} \text{inhibition efficiency (regression equation)} \\ = +72.95 + 7.9A - 7.94B - 3.56C + 5.28AB + 3.30AC - 1.31BC \\ + 2.07A^2 - 1.81B^2 + 10.11C^2 \quad (5) \end{aligned}$$

while corrosion rate (regression equation)

$$\begin{aligned} = 0.017 - 3.940A + 6.050B + 4.950C - 1.13AB \\ + 9.375AC - 8.625BC + 6.455A^2 + 3.095B^2 - 1.205C^2 \quad (6) \end{aligned}$$

The result from ANOVA showed that with Model F-value of 13.68, the model is significant. Furthermore, the significant model terms are A, B, C, AB, C².

Fig. 1 showed plot of Predicted vs Actual values. Fig. 2 showed the graph of Normal plot of residuals vs Internally studentized

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inhibition efficiency



X1 = B: temperature
X2 = C: exposure time

Actual Factor
A: inhibitor conc = 1.00

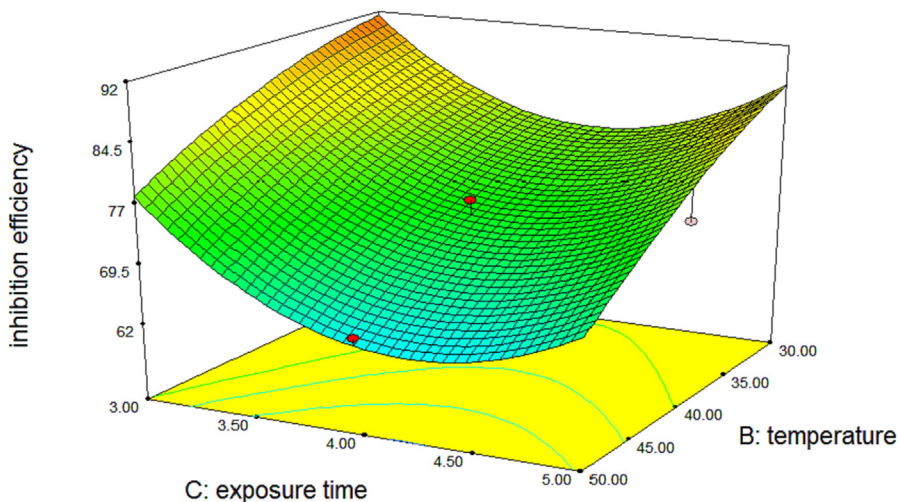


Fig. 3. 3D of Temperature vs Exposure Time.

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inhibition efficiency

Color points by value of inhibition efficiency:

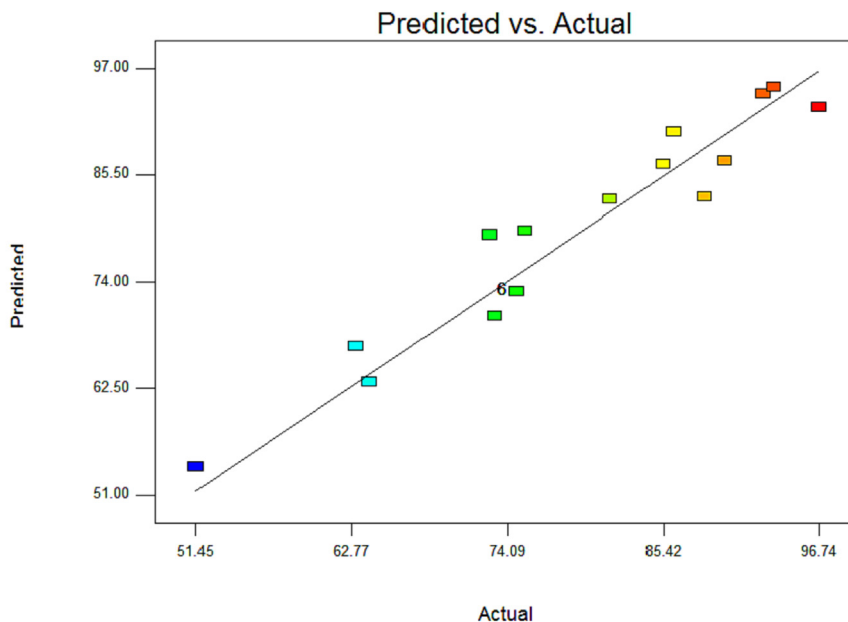


Fig. 4. Plot of the predicted and actual values.

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inhibition efficiency

Color points by value of
inhibition efficiency:

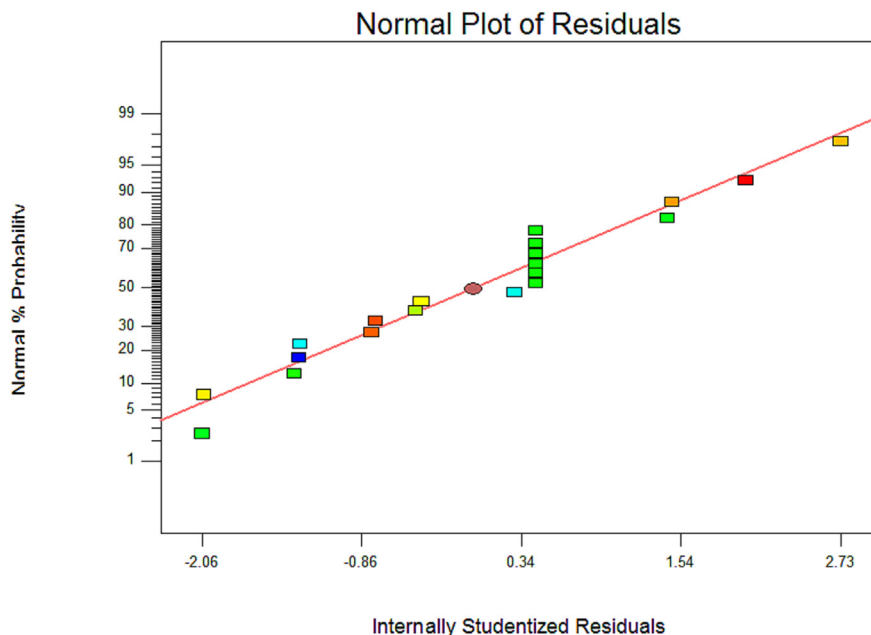


Fig. 5. Internally normal plot of residuals for the inhibition efficiency.

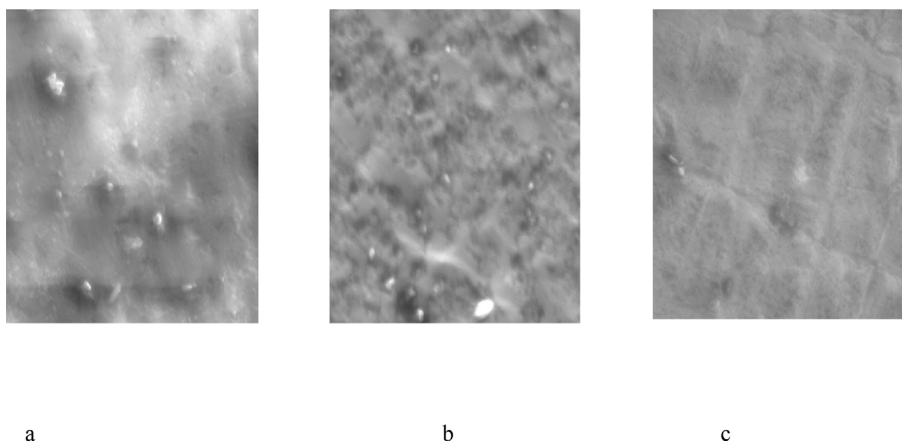


Fig. 6. (a-c): SEM of (a) Blank (b) best process level from experimental (c) coupon of validated experiment.

residuals while Figs. 3–5 showed the 3D surface plots for Inhibition Concentration vs Temperature; Inhibition Concentration vs Time and Temperature vs Exposure time, respectively.

3.4. Result of surface response plots

The 3D Plots are as shown Figs. 1–3. (IE). Fig. 1 illustrated how inhibition effectiveness rises with decreasing time and falls with rising temperature. Fig. 2 showed that as inhibitor concentration increased, inhibitor efficiency decreased over time, indicating that inhibitor efficiency increased with increasing inhibitor concentration. Fig. 3 demonstrated that the metal corrodes faster at higher temperatures and for longer periods of time. The findings correlated with results of Oyewole et al. (2021) [14]; Preethi and Lavanya (2021) [18]; Omran et al. (2022) [19]. Fig. 4 showed the plot of Predicted vs Actual values. Fig. 5 showed the graph of Normal residuals.

3.5. The result of the SEM analysis

The SEM analysis results for the blank, coupon with best process level from experimental design and coupon of validated experiment are shown in Fig. 6a–c, respectively. The coupon in Fig. 6a was greatly damaged while the coupon in Fig. 6b was less damaged because of the presence of the inhibitor. A remarkable improvement on the surface of the coupon in Fig. 6c was observed due to the fact that more film was absorbed on the surface which acted as a barrier and blocked the access of the corrosive species and hence mitigated the corrosion.

4. Conclusion

The results of the phytochemical analysis proved that the extract’s active ingredients were what caused the adsorption and that it was an effective inhibitor. The optimal process variables were: an inhibitor concentration of 1.00 g/l, 4 days of exposure,

and a temperature of 40 °C with an observed inhibition efficiency of 96.74 %; this was based on the experimental design, while the verified experiment showed a result of 97.20 %. The result of the SEM showed that larger protective film had developed on the mild steel of the validated experiment. It can be inferred that PKLE was an effective inhibitor. The result of this research shows promise as it lays the foundation for the use of PKLE as a potential green inhibitor in industrial and oil and gas applications when properly utilized.

Some of the challenges and existing knowledge gap this research addresses are:

- (1) It converts an agricultural waste into a viable product.
- (2) There seems to be scanty literature available on the use of Central Composite Design (CCD) for the interaction and optimization of Palm Kernel Leaf Extract as inhibitor.
- (3) This research will contribute to the Sustainable Development Goal 9 (SGD 9): Industry, Innovation and Infrastructure.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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