

Statistical Analysis of Corrosion Inhibition of Water Hyacinth on Mild Steel in an Acidic Medium

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Abstract

This present work investigated surface response modeling and optimization of corrosion inhibition of water hyacinth on mild steel surface in an acidic medium. This was achieved using the central composite design (CCD) experimental design. Response Surface Methodology was used to assess the effects of experimental process variables that influenced rate of corrosion, and for searching of optimum combinations of factors. The rate of corrosion on mild steel surface was study using weight loss method. The optimum process variables obtained from the quadratic model developed were 1.50 g/l inhibitor's concentration, 8 hours exposure time, and temperature of 60°C with a predicted inhibitor's efficiency value of 82.89%. The experimental result obtained from optimum value validation was 81.5% and the predicted optimum value was adequately represented. The mild steel corrosion inhibition was achieved through the double bond adsorption of carbonyl group existing in the inhibitor phytochemical constituent.

Keywords: Weight loss; Rate of corrosion; FTIR; Phytochemical test; Central composite design; Optimization

Introduction

Corrosion has been described as an irreversible interfacial reaction of a material with its environment destroying useful properties of the material (such as the ductility, electric conductivity and the malleability) by chemical or electrochemical processes with resultant losses including parts replacement, maintenance, repairs, painting, service interruption and loss of product through leakages and contamination [1]. The different techniques used in preventing corrosion, are lubrication, painting, cathodic and anodic protection, electro-painting, material selection, and the use of inhibitors [2]. The use of inhibitors has been widely accepted due to their ability to reduce the rate of corrosion when introduced into a corrosive environment [3]. While literature reports abound in the use of organic and inorganic substances to inhibit corrosion in metals [3-5], there are however, many inorganic corrosion inhibitors with secondary effects in damaging the environment due to their toxicity [3]. Over the years concerted efforts have been made towards replacing these inhibitors with green alternatives which are cheap, eco-friendly, non-toxic, and biodegradable. Researches carried out using plant extracts ranging from papaya leaves, garlic extract, aleos extract to hirta extract to determine their efficiency as corrosion inhibitors on metals and alloys of metals have shown that these plant extracts are efficient corrosion inhibitor on metals in different media [3,6-9].

In recent times, water hyacinth (*Eichhorniacrassipesis*), a water weed and free floating plant that can grow to a height of 3 feet and has persistently constituted a major problem to water ways and aquatic life has been studied as a corrosion inhibitor by researchers and has been found effective in different environments such as acidic environment [10], neutral environment [11], chloride environment [12] and alkaline environment but the use of design of experiment to study the inhibitory action of water hyacinth on mild steel surface to determine the statistical effects of the inhibitory variables on corrosion study have not been used to our best knowledge which necessitated this work. This study therefore seeks to advance the use of Response Surface Methodology (RSM) as a good example of statistical – based approach is a comprehensive experimental design and mathematical modeling, through the partial regression fitting of the experimental factors [13]. RSM is a collection

of mathematical and statistical techniques that are useful for modeling and analysis of engineering problems in which a response of interest is influenced by several variables [14]. It is a technique for designing experiments, building numerical models, evaluating the effects of variables, and searching for optimum combinations of factors [15]. This method is more practical compared to the conventional 'one variable at a time' approach as it arises from experimental methodology which includes interactive effects among the variables and, eventually it depicts, the overall effects of the parameters on the process [16].

Materials and Methods

Materials

The water hyacinth was collected from Agbahro community located in Effurun, Delta State, Nigeria. Soxhlet extractor apparatus, HANNA model pH – 211 (pH meter), Genlab oven model Mino/75/f (oven), weighing balance of model (BH – 600), and beakers, were employed for this corrosion study. Hydrochloric acid, acetic acid, acetone, and ethanol solutions used were of analytical grades and were gotten from a qualified chemical dealer in Effurun, Delta State, Nigeria. Distilled water was procured from the Department of Chemistry Laboratory, Federal University of Petroleum Resources, Effurun Delta, State, Nigeria for sample preparation and solutions.

Methods

Pre-treatment of sample and sample characterization: The water hyacinth was shredded and dried in the oven at 60°C for a period of 48 hours. The water hyacinth particle size range of 600 µm was used for the corrosion study.

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Fourier Transform Infrared Spectroscopy (FTIR): Water hyacinth of 600 µm particle size was observed with FTIR spectroscopy (Buck Scientific model 530) with the range 500-4000 cm⁻¹ (wavelength). The background material used in the analysis is potassium bromate (KBr).

Analysis of the phytochemical constituents in water hyacinth leaf: Screening of phytochemical constituents of water hyacinth leaf extract was carried out to identify the active constituents in the extract. The qualitative and quantitative methods of [17-21] were used for this analysis.

Extraction of water hyacinth leaf extract: 500 g of grounded water hyacinth sample was poured on a muslin cloth. This was then placed inside the 500 ml soxhlet extractor thimble. 200 ml of ethanol was poured into a round bottom flask fixed to the apparatus and the condenser was fixed tightly at the bottom end of the extractor. This set up for the experiment was heated on a heating mantle at 78°C. The ethanol solvent was allowed to remain in contact with the water hyacinth sample for 12 hours while the remaining solvent in the oil was recovered by heating.

Experimental procedure: The corrosion study was carried out using the method described by Nwigbo et al. [22]. The mild steel coupon having dimension of 2 cm × 3 cm × 0.12 cm were polished with abrasive paper, greased to inhibit corrosion, degreased with ether of petroleum and thereafter rinsed with distilled water and dried. The mild steel coupon was suspended with the aid of a thread in 100 ml beaker that contains 100 ml of 0.1 M HCl with various concentrations of the inhibitor. At time interval of 2 hours, 0.1, 0.5, 1.0, 1.5 and 2.0 g/l of inhibitor concentrations of 0.1 M HCl solution, and at various temperatures were studied. Each coupon after corrosion study was inserted in distilled water and dipped in methanol solution which was scrubbed to remove remaining acids, and residual concentration of inhibitors and then washed thoroughly with washing liquor, rinsed with distilled water and thereafter dried in acetone before been reweighed. The experimental plan is as shown in Tables 1 and 2 using design expert software.

Efficiency of inhibitor determination: The efficiency of corrosion inhibition was obtained using the equation below

$$E(\%) = \frac{W_0 - W_1}{W_0} \times 100^3$$

Where, W₀ is the loss in weight in uninhibited medium (blank), and W₁ is the loss in weight in inhibited medium

Experimental design using RSM: The design expert software (Design – Expert 7.00) was used for the experimental runs and modeling of experimental data. Twenty (20) runs of experiment were generated with CCD (Central – Composite Design). The process variables studied were inhibitor concentration (X₁), exposure time (X₂), and temperature (X₃). These three variables were considered at five levels.

Optimization of the RSM Regression Model: Response surface methodology quadratic model generated was optimized using the global response surface equation below;

$$Y = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_{ii} X_i^2 + \sum_{i < j} \beta_{ij} X_i X_j + e \quad (1)$$

For three factor inputs of x₁, x₂ and x₃, the equation of the quadratic response is given as;

$$Y = b_0 + b_1 X_1 + b_2 X_2 + b_3 X_3 + b_{12} X_1 X_2 + b_{13} X_1 X_3 + b_{23} X_2 X_3 + b_{11} X_1^2 + b_{22} X_2^2 + b_{33} X_3^2 \quad (2)$$

Y is the response predicted by the Response Surface Methodology, the linear coefficient is i and j is the quadratic coefficients, regression coefficient is β, while parameters studied is k, and optimized in the experiment, and the random error is e [23].

Results and Discussion

The presence of alkaloids, saponins, terpenoids and aromatic rings in the water hyacinth chemical structure as shown in Table 3 enhanced the process of inhibiting corrosion on mild steel surface. This finding was corroborated by the report of [8,22,24]. Nitrogen and acetylenic alcohols molecules as contained in water hyacinth are adduced by forming a film on metal surface and thus prevent the dissolution process of the mild steel (Anodic reaction) as well as evolution of hydrogen (cathodic reaction) [25].

From Figure 1, the broad band (2655.519-2792.539 cm⁻¹) exhibited aldehyde (C-H) bending. A huge band (C=O) was seen in broad band (1626.748 – 1845.781 cm⁻¹) while alkene (RC=CH₂) was revealed at broad band 923.3814 cm⁻¹. These results were found to be consistent with literature reports of Mshandete et al. [26,27]. The study further revealed that the broad band with frequency (3251.402-3519.133 cm⁻¹) exhibited RO-H (alcohol) wide branded band. Methyne C-H stretch was seen in broad band (2792.539 cm⁻¹) while methylene C-H band was shown in broad band (1462.563 cm⁻¹). The preponderance of carbonyl carbon double bond groups present in the water hyacinth is an indication of its suitability as a useful corrosion inhibitor on surface of mild steel.

Statistical analysis of corrosion inhibition of mild steel using water hyacinth as inhibitor

The ANOVA results depicted in Table 4 shows that the F-value of 35.5 and p-value <0.0001 of the model gotten from mild steel corrosion inhibition using water hyacinth is significant. The experimental data obtained was well represented with the quadratic model obtained and 1.98 value of standard deviation was achieved. R² value of 0.9695 which was considered high was achieved for the model indicating that the predicted value was close to the actual value which suggests it to be accurate [9]. The result from this study indicates that the selected factors was adequately represented by the obtained model and also depict an actual relationship among selected factors. It is seen that 96.95% of the total variation in the inhibitor efficiency of water hyacinth can be

Independent Variables	-α (-2)	Low factor level (-1)	Mid-point factor level (0)	High factor level (+1)	+α (+2)
Concentration of Inhibitor (g/l)	0.1	0.5	1.0	1.5	2.0
Exposure Time, (Hr)	2.0	4.0	6.0	8.0	10
Temperature, (°C)	30	40	50	60	70

Table 1: Central-Composite Design factor levels of independent variables.

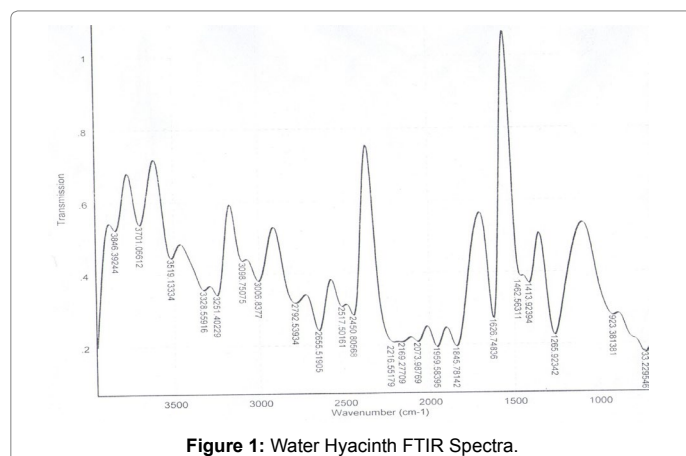


Figure 1: Water Hyacinth FTIR Spectra.

Run Order	Inhibitor Concentration, (g/l)	Exposure Time, (Hr)	Temperature, (°C)
1	1	6	70
2	1	6	50
3	1.5	4	60
4	1	6	50
5	1.5	8	60
6	0.1	6	50
7	1	6	50
8	1	10	50
9	1	6	50
10	1	6	30
11	1.5	4	40
12	1	6	50
13	0.5	8	60
14	0.5	4	60
15	1	6	50
16	1.5	8	40
17	1	2	50
18	2	6	50
19	0.5	4	40
20	0.5	8	40

Table 2: Experimental Design Plan for CCD in Terms of Actual Variables.

Chemical constituents	Percentage composition (%)
Alkaloid	10.40
Terpenoid	5.60
Phenol	Nil
Sterols	Nil
Flavonoid	Nil
Cardiac glycoside	1.80
Glycoside	Nil
Tannin	Nil
Phytosterol	3.40
Saponins	3.20
Anthraquinones	2.60
Reducing sugar	Nil
Phlobatannis	Nil

Table 3: Phytochemical Analysis of Water Hyacinth Leaf Extract.

linked to the experimental variables studied. The significance of each coefficient in the model was checked using the p – values which in turn were useful variables pattern [28]. An adequate precision ratio of 17.132 was obtained in this study which indicates an adequate signal. Results affirmed that there was signal adequate for the obtained model as shown by the value obtained. As depicted in Table 5, the pre R – squared of 0.7427 is in agreement with the Adjusted R – squared value of 0.9421. The coefficient of variation the standard deviation of the mean and experimental data expressed in percentage that is 2.83%, which is lesser than 10% shows that the data from the experiment is reproducible. The significant model terms at 0.05% confidence level were (X_1 , and X_3) linear terms, (X_1X_2 , X_1X_3 , and X_2X_3) the interaction terms and (X_1^2 , and X_3^2) the quadratic terms. This suggests an interaction between the process variables studied which were adjudged to be the core factors that affect the corrosion rate and inhibitors efficiency of a corrosion inhibitor. The RSM model for the corrosion inhibition of mild steel using water hyacinth as an inhibitor was optimize using the desirability function of the RSM. The optimum conditions prediction from the quadratic model were inhibitor's concentration (1.50 g/l), exposure time (8 hours), and temperature (60°C) corresponding to the

inhibitor's efficiency of 82.89%. These were validated with an average inhibitor's efficiency of 81.5% from three replicates and this was closely approximated by the obtained optimum result predicted by the model. The experimentally obtained inhibitor's efficiency is very close to that predicted by the RSM model as seen in Tables 6 and 7 respectively.

The predicted and experimental data inhibitor's efficiency (%) were as well investigated to determine their correlation as depicted in Figure 2. The points of the data on the plot were moderately spread close to the straight line point, an indication of a sound connection concerning the predicted values and experiment data value for the response. This further affirms that the assumptions for the analysis in the study were appropriate for the design space.

Surface response plots

3-D response surface plots of the contour were presented in Figures 3-8. The 3-D surface plots were used to give an important evidence on the system behaviour within the experimental design on the corrosion study. The highest values of the response was credited to the considered factors in the design space as shown by the clear peak in the 3-D response plots. The corresponding plots of contour as represented by the response surfaces projection in the x-y plane provided a clear and very straightforward means of ascertaining the effects of the dependent

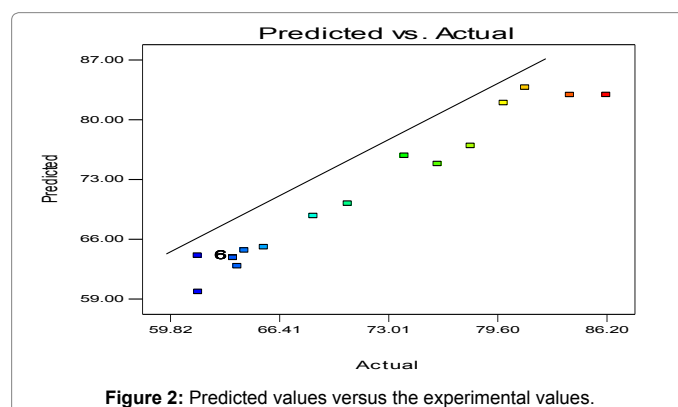


Figure 2: Predicted values versus the experimental values.

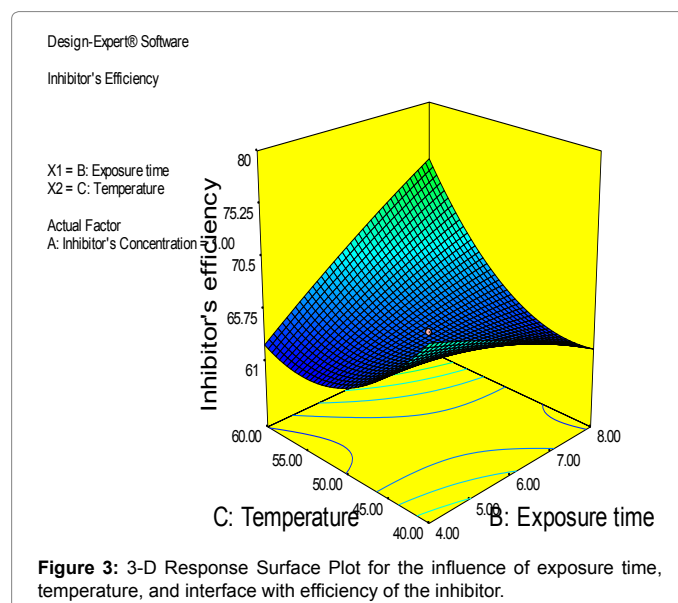
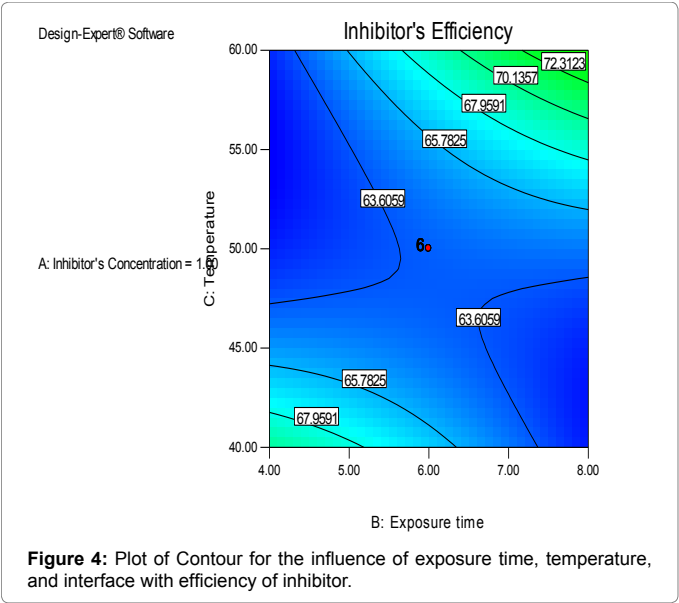


Figure 3: 3-D Response Surface Plot for the influence of exposure time, temperature, and interface with efficiency of the inhibitor.



Parameters Determined	Value
Standard deviation	1.98
Mean	69.86
C.V %	2.83
R - squared	0.9695
Predicted R - squared	0.7427
Adjusted R - squared	0.9421

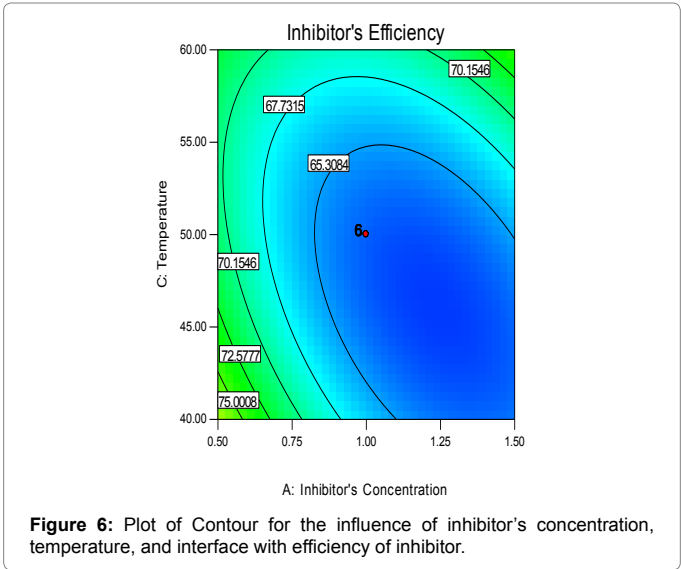
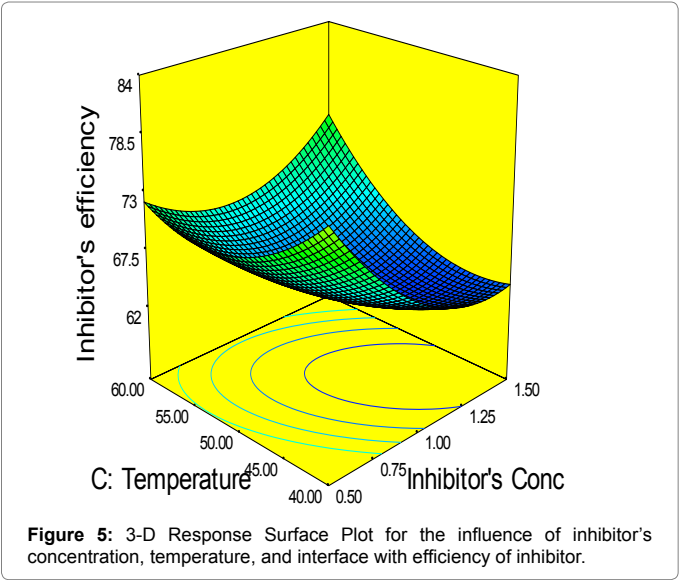
Table 5: Statistical Estimates of CCD.

	X ₁ (Inhibitor's Concentration, g/l)	X ₂ (Exposure Time, Hr)	X ₃ (Temperature, °C)	Inhibitor's Efficiency (%)
Actual Variables	1.5	8.0	60	82.89

Table 6: Result of the Model Optimization.

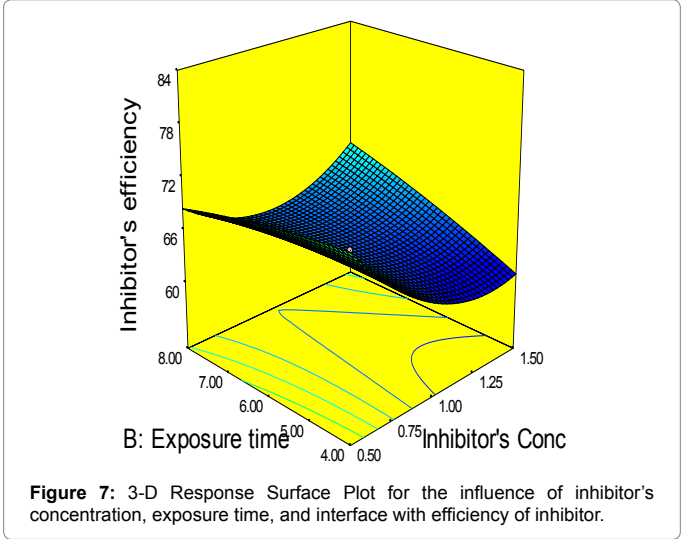
Y ₁ (Inhibitor's efficiency %)	Y ₂ (Inhibitor's efficiency, %)	Y ₃ (Inhibitor's efficiency, %)	Y _{average} (Inhibitor's efficiency, %)
80.00	81.81	82.69	81.50

Table 7: Optimization Validation Result.



Sources	Sum of Squares	Df	Mean Square	F - Values	Prob>F, P - Value
RSM Model	1241.57	9	137.95	35.35	<0.0001 Significant
X ₁	113.55	1	113.55	29.10	0.0003
X ₂	18.02	1	18.02	4.62	0.0572
X ₃	25.25	1	25.25	6.47	0.0292
X ₁ X ₂	69.03	1	69.03	17.69	0.0018
X ₁ X ₃	118.89	1	118.89	30.46	0.0003
X ₂ X ₃	193.65	1	193.65	49.62	<0.0001
X ₁ ²	400.17	1	400.17	102.54	<0.0001
X ₂ ²	5.55	1	5.55	1.42	0.2605
X ₃ ²	384.60	1	384.60	98.55	<0.0001
Residual	39.03	10	3.90	39.03	-
Lack of fit	39.03	5	7.81	39.03	-
Pure error	0.000	5	0.000	0.000	-
Cor Total	1280.60	19	-	-	-

Table 4: ANOVA of Regression Equation Results for Quadratic Model of Corrosion Inhibition Study.



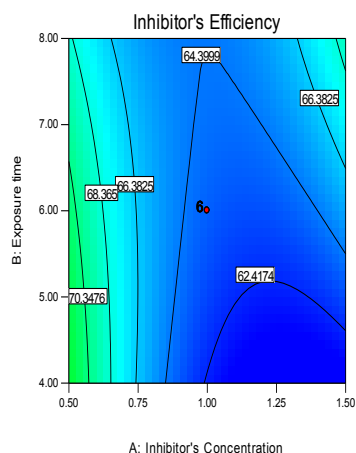


Figure 8: Plot of Contour for the influence of inhibitor's concentration, exposure time, and interface with efficiency of inhibitor.

variable independent variables. The non-circular nature of the contour plots served to indicate effect of interaction effects of the variables studied for the design space within the model.

Conclusion

The existence of saponins, alkaloids, saponins, anthraquinone, flavonoids in the phytochemical screening and the result of the FTIR showing a carbon double bond functional group affirmed that the extract of water hyacinth leaf is a good corrosion inhibitor on mild steel surface. The quadratic model developed using CCD of the response surface methodology revealed that inhibitor's concentration and temperature significantly affected the efficiency of the water hyacinth inhibitor studied.

The optimum process variables obtained from the quadratic model developed were 1.50 g/l inhibitor's concentration, 8 hours exposure time, and temperature of 60°C with a predicted optimum inhibitor's efficiency value of 82.89% using the surface response methodology (CCD) modeling for the experiment design. The results gotten from the validated optimum value was in agreement with that predicted by the quadratic model.

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