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Adsorption Isotherm Analysis of Black Seed (*Nigella Sativa L.*) Oil as an Eco-friendly Corrosion Inhibitor for Mild Steel in Acidic Environment

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ABSTRACT: The objective of this paper is to evaluate the adsorption isotherm analysis of black seed (*Nigella Sativa L.*) oil as an eco-friendly corrosion inhibitor for mild steel in acidic environment using appropriate standard techniques. Weight loss analyses showed that mild steel coupons immersed in the acidic solution without the inhibitor experienced much higher corrosion rates compared to those treated with the extract. Over time, corrosion rates increased initially but began to decline after 72 hours due to protective oxide film formation, consistent with observations from previous studies. Adsorption isotherm models were employed to understand the interaction between the inhibitor and the steel surface. The Langmuir isotherm was determined to be the best fit for the adsorption process, suggesting a monolayer coverage of the inhibitor on the mild steel surface. This model yielded a maximum adsorption capacity (Q_{max}) of 1.03 mg/g and a favorable Langmuir constant (K_L) of 0.24 dm³/g. Scanning Electron Microscopy (SEM) analysis revealed changes in surface morphology, indicating the formation of a protective layer that mitigated corrosion. The mechanism of inhibition is attributed to a donor-acceptor interaction between the inhibitor molecules and iron, leading to a reduction in anodic and cathodic reactions. In conclusion, black seed oil extract demonstrates significant potential as an eco-friendly corrosion inhibitor for mild steel in acidic conditions, providing a sustainable alternative to conventional synthetic inhibitors.

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Corrosion of metals is a widespread issue that poses significant challenges due to its harmful effects on structures and equipment (Mohammed *et al.,* 2022). The International Union of Pure and Applied Chemistry (IUPAC) characterizes corrosion as a material interaction that is irreversible and influenced by environmental conditions, leading to the degradation or dissolution of materials (Holla *et al.,* 2024). With growing environmental concerns, there is a pressing need for safe and non-toxic alternatives to synthetic corrosion inhibitors, as many regulations now limit their use (Mohsen *et al.,* 2024). Corrosion is an undesirable process that not only compromises the integrity and aesthetics of materials but also shortens their operational lifespan (Mohammed *et al*., 2022). The impact of corrosion extends beyond mere material damage, affecting environmental health, human safety, and industrial efficiency (Mohsen *et al.,* 2024). Mild steel, with its low carbon content (under 0.25%), is one of the most widely used forms of steel due to its

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affordability, strength, ductility, ease of welding, and adaptability for various applications (Udoisoh *et al.,* 2024). Corrosion inhibitors are compounds that help decrease the rate of corrosion in metals exposed to aggressive environments by disrupting anodic, cathodic, or both electrochemical reactions on the metal surface (Pramanik *et al.,* 2022). Research aimed at mitigating the adverse effects of carbon steel corrosion is essential due to its potential risks to the industrial sector (Mohsen *et al*., 2024). Various strategies have been developed to safeguard the material from corrosion, including cathodic protection, protective coatings, and the use of corrosion inhibitors, however, these methods often have limited effectiveness due to factors like cost and the difficulties associated with modifying materials (Mohsen *et al.,* 2024; Rao and Mulky, 2023).

Originally, *Nigella Sativa L*., commonly known as black seed, is obtained from a flowering plant recognized for its culinary, preservative, and medicinal applications throughout history. It has recently gained attention as a valuable source of edible oil (Alrashidi *et al*., 2020). The oil derived from black seeds is renowned for its wide range of pharmacological properties, including antiparasitic, antihypertensive, analgesic, antineoplastic, and antibacterial effects. These benefits contribute to its efficacy in mitigating hepatotoxicity and nephrotoxicity, largely due to the presence of various active compounds (Alrashidi *et al.,* 2020; Udoisoh *et al*., 2024). Given these promising attributes, there is a growing interest in investigating how corrosion inhibitors, particularly those derived from Nigella sativa, interact with mild steel. Researchers are focusing on developing advanced techniques and models to better understand the inhibitive mechanisms and overall effectiveness of these natural inhibitors (Abkedi *et al.,* 2024; Udoisoh *et al*., 2024). Hence, the objective of this paper is to evaluate the adsorption isotherm analysis of black seed (*Nigella Sativa L*.) Oil as an eco-friendly corrosion inhibitor for mild steel in acidic environments.

MATERIALS AND METHODS

Extraction of oil from Black seed oil: The soxhlet extractor was utilized for this process, Black seeds were collected from central market Kaduna, Nigeria, which were later ground into finer particles, from which 100 grams were obtained and loaded into the soxhlet apparatus, where hexane was used as the solvent for extraction and refluxed for 4 hrs. A rotary evaporator was utilized for the final separation, the extracted oil was stored in an amber bottle (Rahim *et al.,* 2022).

Surface characterization using HRSEM: To better understand the surface morphology of the mild steel, the samples were analysed through HRSEM analysis before and after inhibition using the Polaron SC515 scanning electron microscope (SEM) at an acceleration voltage of 20 kV (Mohsen *et al.,* 2024).

Metal Preparation: The mild steel sheets used for this study were sourced locally in Kaduna, Nigeria, where each sheet was mechanically press cut into 4 cm x 4 cm coupons at a thickness of 0.04 cm. They were then degreased in absolute ethanol and further dried in acetone and then stored in a desiccator before use (Abakedi *et al.,* 2024).

Preparation of Corrosive medium: Hydrochloric acid (HCL) was prepared at a concentration of 1 M, where deionized water was used for preparation of the solution (Alamry *et al.,* 2023).

Corrosion Inhibition Study: For this process, the gravimetric method was utilized, here, the effect of immersion time and concentration of the corrosion inhibitor (black seed oil) on the mild steel was investigated. In the first series of experiments, individual mild steel coupons were fully immersed in beakers containing 50 mL of 1M HCl solution, with 4 mL of black seed oil added as an inhibitor. The weight loss of the coupons was measured at 24-hour intervals over a period of 5 days. In a separate experiment, the weight loss of the coupons with and without the inhibitor was monitored over a 48-hour period, here, only the concentration of Black seed oil was varied (2 mL – 10 mL), this is to determine the effects of the concentration of the oil extracts of black seed oil on the corrosion inhibition of mild steel. In each experiment, the coupons were dipped in acetone, they were then brushed gently using a sandpaper, washed with deionized water, wiped with tissue paper and then dried. From the weight loss values, corrosion rates were computed accordingly using equation 1.

$$\mathrm{CR}\ (\mathrm{g/cm} - 2\mathrm{h} - 1) = \frac{\mathrm{w1} - \mathrm{w2}}{At} \qquad \mathbf{1}$$

Where CR is the total corrosion rate, w1 and w2 are the initial and final weights respectively, A is the cross sectional area and t is the exposure time.

The inhibition efficiency (Ƞ %) of black seed oil was evaluated from the following equation:

$$\Theta = \frac{\mathrm{CR\ (blank) - CR\ (inh)}}{CR\ (blank)} \qquad \mathbf{2}$$

$$\eta\ (\%) = \frac{\mathrm{CR\ (blank) - CR\ (inh)}}{CR\ (blank)} \times 100 \qquad \mathbf{3}$$

Where Ɵ is the surface coverage, CR (blank) and CR (inh) are the corrosion rates of the mild steel coupons in the absence and presence of an inhibitor, respectively (Mohammed *et al.,* 2022).

RESULTS AND DISCUSSION

Composition of Mild steel: The XRF analysis carried out shows the chemical composition of the mild steel sample utilized for this study. The iron content was 98%, carbon was 0.21%, Manganese was 1.0% while that of Silicon, Sulfur, Phosphorus and copper were found to be 0.24%, 0.06%, 0.45% and 0.04 respectively. Mild steel is often used for industrial applications due to the high iron content, this is what makes it susceptible to corrosion.

Weight Loss Analysis: The corrosion rate variation with increasing immersion time is presented in both Table 1 and Table 2. Table 1 shows the results obtained in the absence of the inhibitor, while Table 2 highlights the effect of time variation in the presence of the corrosion inhibitor. In both cases, the mild steel coupons immersed in the acidic solution without the inhibitor exhibited a higher corrosion rate at each time interval compared to those in the inhibitor-containing acidic solution (Table 2).

Table 1: Variation of rate of corrosion in 1M HCL with time in the absence of the corrosion inhibitor

Coupon	Immersion Time	DW	CR
A	24	0.09	0.000234
B	48	0.21	0.000273
C	72	0.36	0.000313
D	96	0.51	0.000332
E	120	0.65	0.000339

It was generally observed that the corrosion rate increased as the immersion time extended from 24 hours to 48 hours; however, the corrosion rate started to decline as the immersion time went from 72 hrs to 120 hrs especially in the case of the coupons immersed in the media containing the inhibitor. These observations re very similar to that of Alamry *et al.* (2023) and Mohammed *et al.* (2022), these variations from 72 hrs – 120 hrs are attributed to the fact that oxide films having protective properties tend to form on the surface of the metal being corroded, which tends to stabilize the material, however, they get destroyed when the immersion time surpasses 120 hrs due to tension (Alamry *et al.,* 2023).

Table 2: Variation of rate of corrosion in 1M HCL with time in the presence of 4 mL of the corrosion inhibitor

Coupon	Immersion Time (hrs)	DW	CR
F	24	0.076	0.000198
G	48	0.15	0.000195
I	72	0.22	0.000191
J	96	0.25	0.000163
K	120	0.26	0.000135

From table 3, it can be observed that corrosion rate of the mild steel increased with increase in concentration of the inhibitor, this is because the molecules of the extract get absorbed on the surface of the metal which favors anodic polarization (Mohammed *et al.,* 2022).

Table 3: Corrosion rates and inhibition efficiencies of steel rods in 1M HCL with varying concentration of the corrosion inhibitor

C (mL)	CR	Ɵ	Ƞ (%)
2	0.000115885	0.258333	25.83333
4	0.000208333	0.238095	23.80952
6	0.000325521	0.21875	21.875
8	0.000494792	0.24	24
10	0.000690104	0.253521	25.35211

Adsorption isotherms are very important models used to better understand the interaction between the inhibitor and the surface of the mild steel as it is preventing corrosion. The figs. 1-3 are isotherms depicting the Langmuir, Freundlich and Temkin plots for the inhibition process of mild steel in the presence of black seed oil which served as the inhibitor.

Isotherm studies: The results obtained from the gravimetric studies was used to select the most appropriate isotherm for the adsorption process of the inhibitor solution on the surface of mild steel. R^2 being the linear regression coefficient, proved that Langmuir isotherm is the best fit.

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \qquad 4$$

Where Ɵ represents the fraction of the surface covered by the adsorbed inhibitor molecules and C is the concentration of the inhibitor. Here, the equilibrium adsorption constant is denoted by K_{ads}, this can be obtained from the intercept value of the plot of $\frac{C}{\theta}$ against C (Alamry *et al.* 2023).

Table 4: Isotherm parameters for the adsorption of inhibitor molecules on the surface of mild steel

Langmuir	
Q_{max} (mg/g)	1.03289779
K_L (dm^3/g)	0.242224
R_L	0.6736511
R^2	0.98453
MSWD	3.34617
Freundlich	
K_f (L/mg)	0.25287739
1/nf	-0.02831
R^2	0.07647
MSWD	0.00098285
Temkin	
B_T (J/mol)	-0.01601
K_T (L/mg)	1.35E-07
R^2	0.08147
MSWD	2.94E-04

Langmuir adsorption is concerned with surface coverage and balancing of the rate of adsorption and desorption, as adsorption is proportional to that portion of the surface of the mild steel that is open whereas desorption is proportional to the portion of the mild steel surface that is covered. S seen in fig. 1, this model aids in estimating the maximum adsorption capacity as well as understanding whether or not the adsorption process which occurred in the homogeneous binding sites was at a homogeneous monolayer (Ragadhita and Nandiyanta, 2021). The Langmuir constant is denoted by K_L, while the separation factor (dimensionless constant) is denoted by R_L. The correlation coefficient of the adsorption process is denoted by R^2, Q_{max} describes the materials maximum capacity for adsorption. As shown in Table 4, the materials have a great affinity for the removal of MB, this is because of the high Q_{max} at 1.03289779. With a Langmuir separation factor (R_L) of 0.6736511, mild steel appears to have a favorable adsorption process. Adsorption is unfavorable when R_L is above 1, liner when R_L is equal to 1 and strong when R_L is equal to 0. The Langmuir constant K_L on the other hand represents affinity between the inhibitor and the mild steel surface, calculated as 0.242224 dm^3/g.

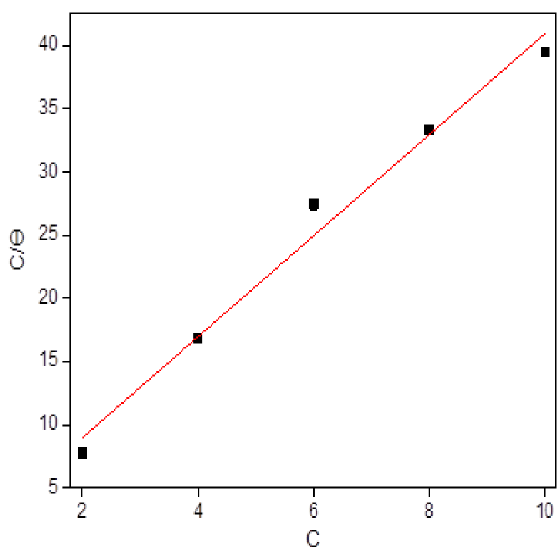

Fig. 1: Langmuir adsorption Isotherm

A higher magnitude of K_L means there is a lot more affinity between the mild steel and corrosion inhibitor. A higher value of K_L indicates a strong interaction while a smaller K_L value indicates a weak interaction between the inhibitor molecule and the surface of mild steel. In addition, R^2 which represents the regression coefficient, SSE which is the sum of squares errors for the analysis, and lastly, MSWD which represents the mean squared weighted deviation (MSWD) indicate the suitability of an isotherm model for the entire adsorption process where the highest R^2 and lowest SSE and MSWD are used to determine the best fit isotherm model (Dev *et al.*, 2022). Based on the values obtained for R^2 and error analysis (MSWD) presented in table 4, the langmuir isotherm at 0.98453 and 3.34617 respectively, the Langmuir isotherm fitted well with regards interpreting the interactions occurring during the adsorption process. (Abubakar *et al.*, 2023; Ragadhita and Nandiyanta, 2021).

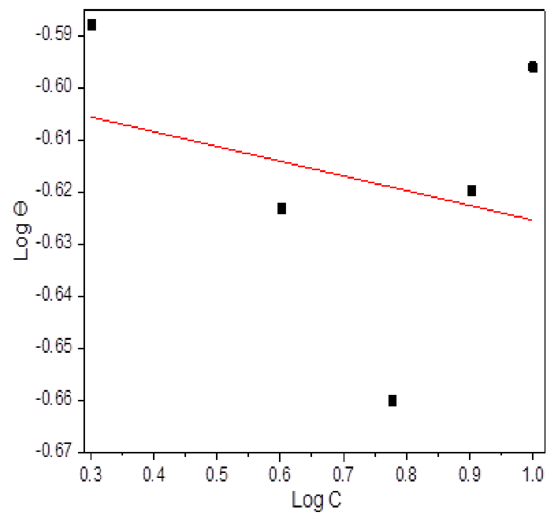

Fig. 2: Freundlich adsorption Isotherm

Freundlich isotherm model is used to explain a physical type of adsorption where adsorption occurs in several layers, this isotherm as seen in fig. 2, is based on the assumption that the adsorption sites are heterogenous. K_F is known as the Freundlich constant, n is the value which indicates the degree of linearity between the inhibitor solution to the heterogeneity of the surface. From the results of the Freundlich isotherm obtained from the fitting data, the adsorption parameters were determined. From Table 4, the value of K_F is 0.25287739 L/mg. Linear adsorption is present when $n_F = 1$, however, when $n_F < 1$ it means that the adsorption process is a favorable chemical process., On the other hand when $n_F > 1$, physisorption is occurring, and a favorable adsorption is only possible when $0 < 1/Nf < 1$, a cooperative process of adsorption is occurring when $1/n > 1$. However, for this interaction the value gotten was -0.02831. According to Table 4, the R^2 value was 0.07647 which is extremely low, while the MSWD for the interaction was calculated as 0.00098285. Hence, it is unlikely that multilayer coverage occurred on the mild steel (Abubakar *et al.*, 2023).

Temkin isotherm is based on three postulates, the heat of adsorption decreases as the surface mild steel coverage increases, existence of distribution of a uniform binding energy on the surface of the mild steel and that the adsorption process involves the interaction between the inhibitor-mild steel. Here, B_T represents the adsorption heat constant if $B_T < 8$ KJ/mol, then the adsorption process occurs physically and if $B_T > 8$ KJ/mol, chemisorption process occurred, here, B_T is equal to -0.01601 KJ/mol, meaning adsorption occurred physically. The binding equilibrium constant also known as the adsorption potential for the Temkin isotherm is represented by K_T, while the absolute temperature is represented by T.

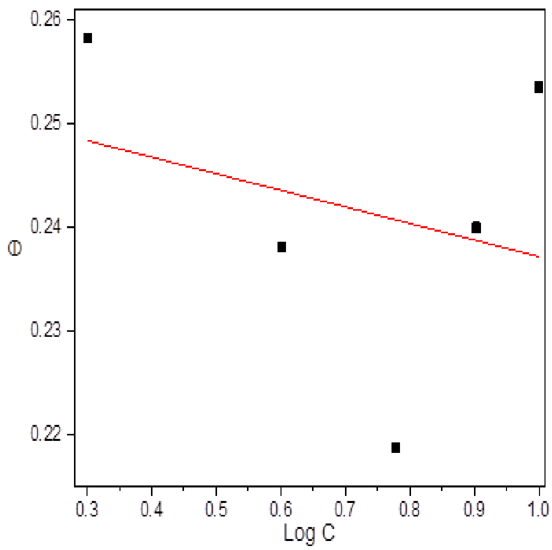

Fig. 3: Temkin adsorption Isotherm

A high value for K_T displays attractive interaction between the inhibitor-mild steel system, 0.00000013522 L/mg was the value calculated for K_T. These values are quite low, which implies that the affinity between the mild steel and the inhibitor molecules is not much. In the end, the lower values for B_T properly is an indication of the good interaction taking place between the mild steel and indictor while in the acidic environment. The R^2 value is 0.08147 which is less than that of Langmuir, meaning data is not suitable for Temkin model. In addition, the MSWD was calculated as 0.00029362. Hence, the intersection between the inhibitor and mild steel cannot be described by this particular isotherm (Abubakar *et al.*, 2023; Ragadhita and Nandiyanta, 2021). Based on the R^2 value for each adsorption model, the adsorption system in $ZnWO_4$ is compatible with Langmuir only. Hence, the adsorption system forms a monolayer inhibitor on the surface of the mild steel, based on the assumption of the Langmuir model.

Surface Morphology: SEM micrographs at 100 nm were used to analyse the morphological variation of the mild steel sample with exposure to the inhibitor in the 1M HCL environment. After polishing, the surface of the mild steel was defect free as seen in plate 1, however upon contact with the inhibitor environment, some roughness was observed, see plate 2, and this is because parts of the metal surface were beginning to dissolve. This is due to the presence of protective layer formed by the inhibitor as seen in plate 3. This layer prevented the surface from any roughness, this proves further that the occurrence of black seed oil on the surface of mild steel can help decrease the rate of corrosion (Alamry *et al*., 2023).

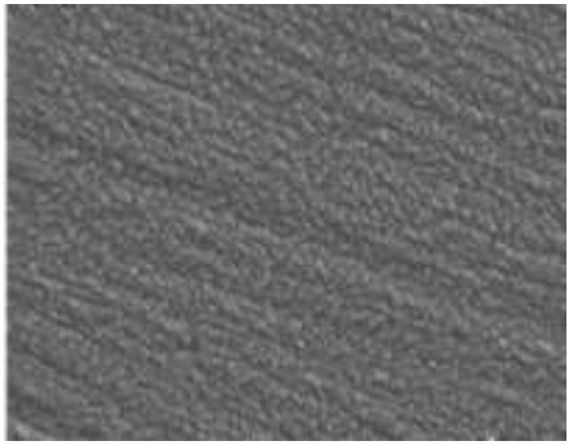

Plate 1: SEM Micrographs of Mild Steel surface before interaction

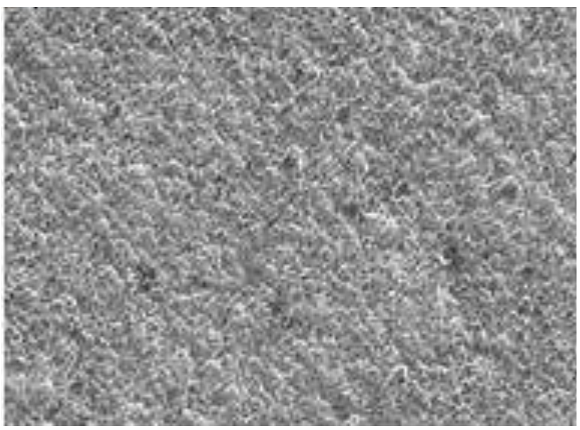

Plate 2: SEM Micrographs of Mild Steel surface immersed in 1M HCL in the presence of the inhibitor

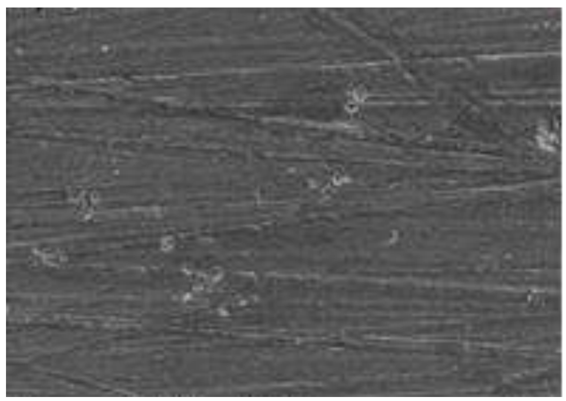

Plate 3: SEM Micrographs of Mild Steel surface after immersion in 1M HCL in the absence of the inhibitor.

Mechanism of Corrosion Inhibition of Mild Steel in 1M HCL by black seed oil: In an acidic environment such as 1M HCL, the surface of mild steel tends to become positively charged due to protonation by hydrogen ions. The molecules from the oil extract, which can exist either in neutral or protonated forms, interact with the steel surface through a donor-acceptor mechanism. This involves the transfer of electron density from the electron-rich atoms (such as oxygen, nitrogen, or π-electron systems) present in the inhibitor to the vacant d-orbitals of iron (Fe) atoms on the steel surface. This electron donation leads to the formation of coordinated bonds, anchoring the inhibitor molecules onto the steel surface. This adsorption process results in a protective film formation, which reduces the metal's contact with the corrosive environment. The inhibitor's action is dual in nature, as it suppresses both the anodic (metal dissolution) and cathodic (hydrogen evolution) reactions. This comprehensive blocking of electrochemical reactions effectively decreases the rate of corrosion. As a result, black seed oil extract proves to be an efficient and environmentally friendly corrosion inhibitor, offering a green alternative to conventional synthetic inhibitors (Jayakumar *et al.*, 2024).

Conclusion: This study demonstrates that black seed oil extract acts as an efficient green inhibitor for mild steel in a 1M HCl acidic environment. Weight loss measurements revealed a significantly higher corrosion rate without the inhibitor, while the presence of the extract substantially reduced corrosion by creating a protective layer on the steel surface. The adsorption data corresponded most closely to the Langmuir isotherm model, indicating that the inhibitor molecules form a monolayer on the mild steel through donor-acceptor interactions.

Declaration of Conflict of Interest: The authors declare no conflict of interest.

Data Availability Statement: Data are available upon request from the first author or corresponding author.

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