

## **Investigation of the Behavior of Aminated Jojoba Derivatives as Green Corrosion Inhibitors for Mild Steel at 0.5 N HCl**

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### **ABSTRACT**

*Jojoba oil was brominated using bromine, then three aminated jojoba derivatives were synthesized via amination of the brominated jojoba oil, with different types of amines (aniline; 2, 4-dimethyl aniline, and 4-amino benzoic acid). The prepared compounds were elucidated using FT-IR and <sup>1</sup>H-NMR. The behavior of the aminated jojoba derivatives was investigated as corrosion inhibitors at different temperatures (308, 318, 328 and 338K) for mild steel using 0.5N HCl via weight loss method, and chemical analysis method. The prepared compounds exhibit anti-corrosion properties for mild steel at acidic medium; while corrosion inhibitor II based on 2, 4- dimethyl aniline exhibits an excellent corrosion protection property.*

**Keywords:** Aminated jojoba oil, Corrosion inhibitors, Mild steel, Weight loss method, Chemical analysis method

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### **INTRODUCTION**

Jojoba plant is well known in botanical literature as *Simmondsia Chinensis* [1-4]. It is a desert shrub that grows wild in Southern Arizona, north-western Mexico and neighboring areas. Greene and Foster [5] were the first to report that jojoba nuts contained about 46% of liquid oil which resembled sperm whale oil in its analytical characteristics and they are considered to be a liquid wax, which consist mainly of fatty acid esters of decyl alcohol, that contain two double bonds in each constituent molecule.

The potential importance of jojoba oil for industrial uses has stimulated research. Jojoba oil has been endorsed at the field of cosmetics; such as hair oil, creams, shampoo, conditioning rinse. Due to its higher thermal and oxidative stability, scientists all over the world increased their scientific searches on the industrial applications of jojoba oil. Jojoba oil has been used as lubricant [6-8], additives for lubricant [9,10], surfactant [11], micro-emulsion [12], antioxidants [13], corrosion inhibitors [14], friction modifiers [15], viscosity index improvers and pour point depressants [16-19], extreme pressure additives [20], bio-fuel [21-23], and bio-diesel [24-26].

In the present work, novel green aminated derivatives of jojoba oil were synthesized via brominating of jojoba oil using bromine, then amination of the brominated jojoba derivative using different types of amines. The prepared amines were investigated as corrosion inhibitors for mild steel at acidic medium, using different intervals of temperatures.

### **MATERIALS AND METHODS**

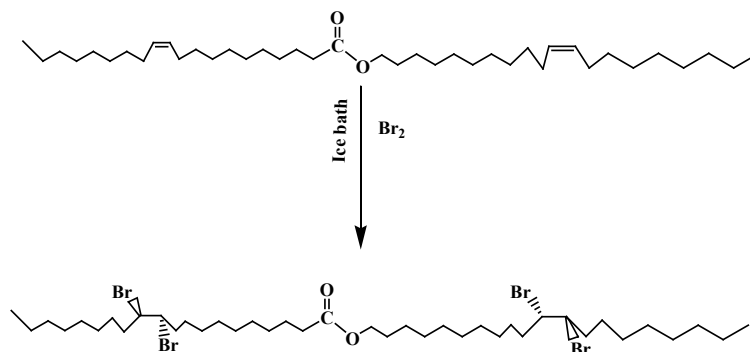
#### **Materials**

Jojoba oil was purchased from Dreams Mill for oils; Saudi Arabia, bromine, aniline, 4- amino benzoic acid and 2, 4-dimethyl aniline, were purchased from Aldrich.

## Procedures

### *Brominating of Jojoba Oil*

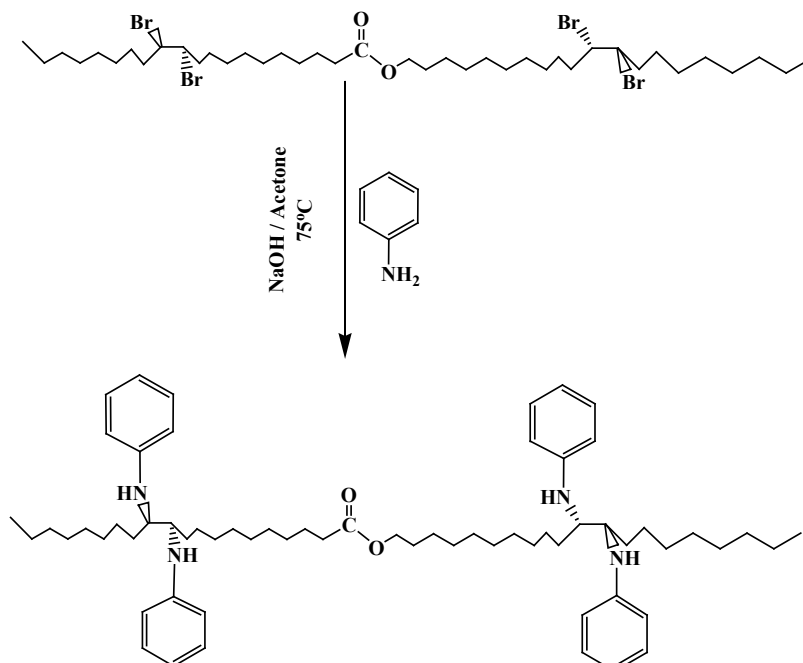
In an ice bath a 250 ml round bottom flask containing (1 mole of jojoba oil, 32.4 g) was connected directly to a dropping funnel containing 2 moles of bromine (31.96g). The bromine was allowed to the reaction drop by drop till the end of addition (the color of the oil changed from yellow to dark brown), **Scheme 1**.



**Scheme 1:** Brominated jojoba.

### *Amination of brominated jojoba with different types of amines*

In 250 ml round bottom flask, about 1 mole of brominated jojoba (32.18 g) was added to 4 mole of [aniline (18.6 g), 2,4-dimethyl aniline (24.2 g), and 4-amino benzoic acid (27.4 g)] separately, 10 ml of Acetone, and 3 ml of 0.5N NaOH solution. Connect the flask directly to condenser and the reactants were allowed to condense at 75°C for one hour, **Schemes 2-4**. The products (J<sub>1</sub>-J<sub>3</sub>) were washed after cooling with distilled water separately, to remove the excess NaOH solution, and then wash with acetone. **Table 1**, indicates the designation and the chemical composition of the prepared compounds.

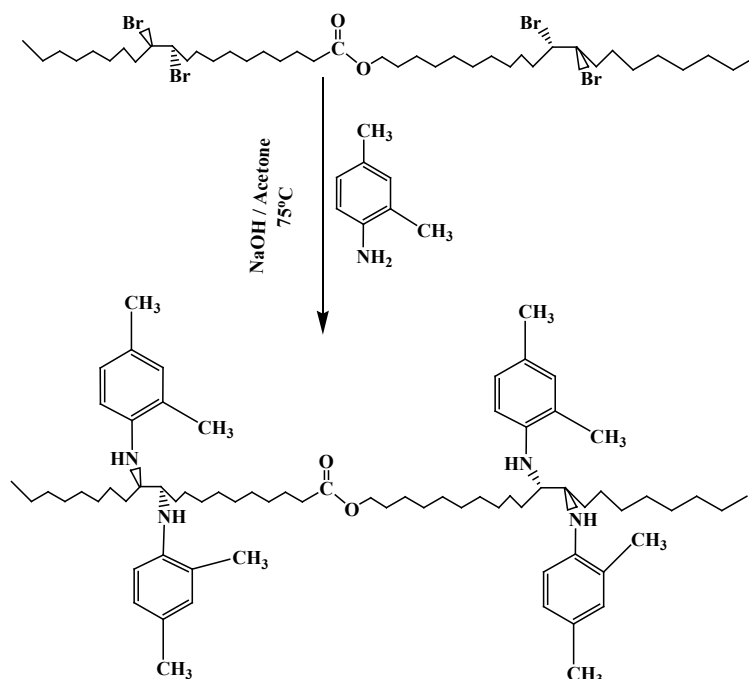


**Scheme 2:** Aminated jojoba derivative with aniline.

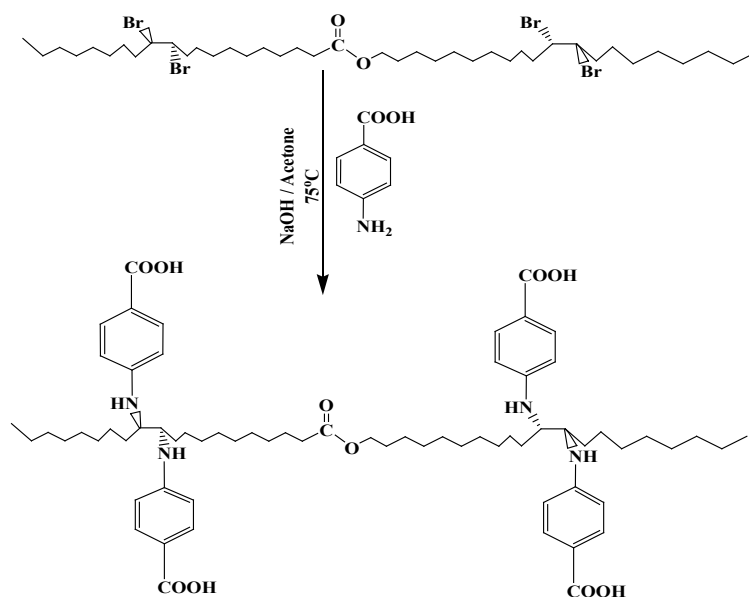
### **Elucidation of chemical structure of the prepared compounds**

#### *Using FT-IR*

The prepared compounds were elucidated using Nicolet iS10-FTIR Spectrophotometer, KBr, Jazan University, Saudi Arabia



Scheme 3: Aminated jojoba derivative with 2,4 di-methyl aniline.



Scheme 4: Aminated jojoba derivative with 4 - amino benzoic acid.

Table 1: Chemical composition of the prepared ( $J_1 - J_3$ ) compounds

| Designation | Composition                                  |
|-------------|--|
| $J_1$       | Brominated jojoba with aniline               |
| $J_2$       | Brominated jojoba with 2,4-di methyl aniline |
| $J_3$       | Brominated jojoba with 4-amino benzoic acid  |

**Using  $^1\text{H-NMR}$** 

The structure of the prepared compounds were elucidated using Proton Nuclear Magnetic Resonance " $^1\text{H-NMR}$ " spectra using a JEOL NMR ECA-500 using DMSO as a solvent; NRC, Dokki, Egypt.

## Corrosion studies

### Weight Loss Measurements

Mild steel coupons (CS37) having chemical composition mentioned in **Table 2**, with dimensions  $7.0 \times 5.0 \times 0.3$  cm were abraded using different grades of emery papers grades (310, 410, and 610), washed with bi-distilled water, degreased with acetone, dried and kept in a desiccator, weight accurately using a digital balance with high sensitivity. The coupons were immersed in 0.5N HCl solution with and without various concentrations (250, 500, 1000, 2000, and 3000) ppm of the prepared inhibitor for 3 hours, at different temperatures (308, 318, 328 and 338K). Weight loss experiments were carried out according to the **ASTM G31 - 72** standard procedure described in reference [27]. In brief, carbon steel specimens in triplicate were immersed in 100 ml 0.5N HCl containing various concentrations of the studied inhibitors. The mass of the specimens before and after immersion was determined using an analytical balance accurate to 0.01 mg. Reading were taken after each one hour, the coupons were washed with bi-distilled water, dried and weighed accurately [28-35]. The investigations carried out in the open air. For further data processing, the average of the three replicate values was used. The pre-cleaned and weighed coupons were suspended in beakers containing the test solutions using glass hooks and rods. The weight loss was calculated by equation (1):

$$\Delta W = W_1 - W_2 \quad (1)$$

**Table 2:** Chemical composition of mild steel (CS-37) alloy

| Element   | C    | Mn   | P    | S    | Si   | Fe    |
|-----------|------|------|------|------|------|-------|
| Ratio (%) | 0.11 | 0.45 | 0.04 | 0.05 | 0.25 | 99.10 |

Where  $W_1$  and  $W_2$  are the weights of coupons before and after immersion.

The surface coverage area ( $\theta$ ) for the different concentrations of the prepared corrosion inhibitors in 0.5N HCl was calculated using equation (2):

$$\theta = 1 - (W_1/W_2) \quad (2)$$

The inhibition efficiency according to weight loss method  $IE_w$  (%) was determined by equation (3):

$$IE_w \% = (W_{corr} / W'_{corr}) \times 100 \quad (3)$$

Where  $W_{corr}$  and  $W'_{corr}$  are the corrosion rate of coupon with and without corrosion inhibitors, respectively.

The corrosion rate (CR) was calculated by equation (4):

$$CR = (W_1 - W_2) / At \quad (4)$$

Where  $W_1$  and  $W_2$  are the weight of coupons before and after immersion in test solutions. A is the total area of the coupon ( $cm^2$ ), and t is the immersion time (h).

### Chemical Analysis of Solution according to weight loss method

When a metal undergoes corrosion in an electrolyte of a fixed volume, the cations of corroding metal will accumulate in solution. Accordingly, the solution becomes more concentrated in the dissolved cations with the progression of time [36]. Thus, chemical analysis of withdrawn aliquots of the solution as a function of time allows determination of the corrosion rate according to equation (5).

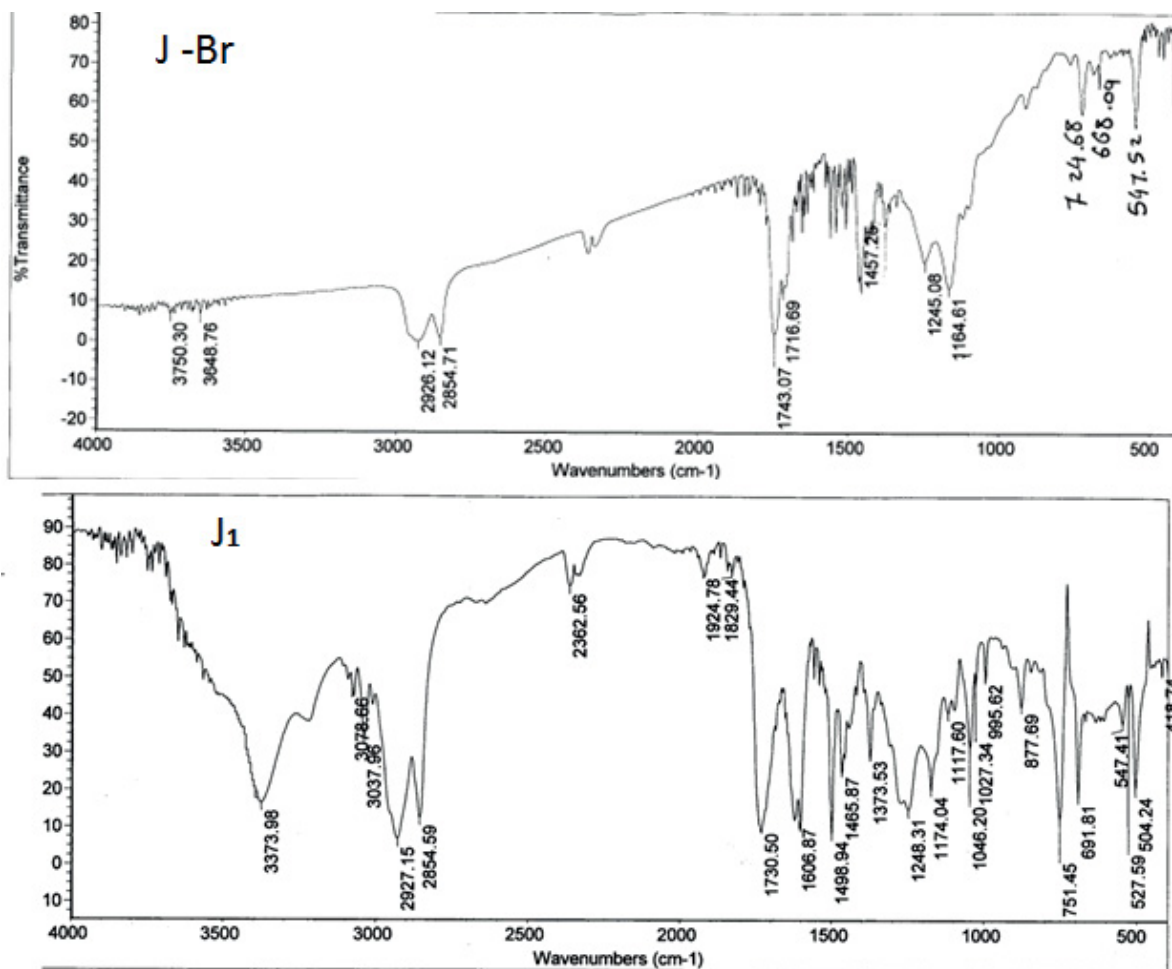
$$CR_{Fe} \left( \frac{g}{cm^2 \cdot h} \right) = \left( \frac{[Fe+3] \text{ mole}}{cm^2 \cdot h} \right) \left( \frac{1 \text{ mole Fe}}{1 \text{ mole } [Fe+3]} \right) \left( \frac{55.85 \text{ g Fe}}{1 \text{ mole Fe}} \right) \quad (5)$$

## RESULTS AND DISCUSSION

Electrophiles that have leaving groups (X) attached to C ( $sp^3$ ) usually undergo substitution or elimination reactions. Nucleophilic substitution at carbon has received exceptionally detailed mechanistic study by organic chemists. The reaction is of broad synthetic utility, and many individual observations as accumulated before systematic efforts to characterize the reaction mechanism began. In a nucleophilic substitution reaction, a nucleophile- electrophile  $\sigma$  bond replaces the electrophile  $-X\sigma$  bond. Substitution reactions at  $1^\circ C$  and  $2^\circ C$  ( $sp^3$ ) usually proceed by the  $S_N2$  mechanism under basic or neutral conditions. In  $S_N2$  mechanism, the nucleophile  $Nu^-$  approaches the electrophilic center opposite X and in line with the C-X bond. The lone pair on  $Nu^-$  is used to form the C-Nu bond, and the pair of electrons in the C-X bond simultaneously leaves with X as the bond breaks. The other three groups on C move away from Nu and toward X as the reaction proceeds, so that when the reaction is complete, the stereochemistry of C is inverted [37].

### Elucidation of the prepared compounds

The prepared brominated jojoba derivative (J) was elucidated using Fourier Transform Infrared Spectroscopy (FT-IR), **Figure 1**. One can noticed the disappearance of (C=C) peak at  $1638\text{ cm}^{-1}$  and appearance of peak corresponding to C-Br bond at  $725\text{ cm}^{-1}$ , this confirm the complete addition at C=C bond. FT-IR spectrum of  $J_1$  compound as an example of the prepared aminated jojoba derivatives indicates the disappearance of C-Br bond at  $725\text{ cm}^{-1}$  and appearance of one peak at  $3374\text{ cm}^{-1}$  corresponding to NH group. The presence of a peak at  $1730\text{ cm}^{-1}$  insures the stability of the ester group toward amination reaction.  $^1\text{H-NMR}$  analysis of brominated jojoba (J) and aminated jojoba derivatives ( $J_1$ - $J_3$ ) are mentioned at **Table 3**.



**Figure 1:** FT-IR of brominated jojoba and aminated jojoba derivative with aniline.

### Corrosion tests

#### Weight loss method

The inhibition efficiency of the organic compounds depends on many factors including the number of adsorption sites and their charge density, molecular size, heat of hydrogenation, mode of interaction with the metal surface and formation of metallic complexes [38]. Weight loss parameters were mentioned at **Table 4**.

#### Effect of ( $J_1$ - $J_3$ ) concentrations

The variation of inhibition efficiency ( $IE_w\%$ ) from weight loss measurements with ( $J_1$ - $J_3$ ) concentrations is shown in **Figure 2**, while the weight loss parameters; corrosion rate CR, surface coverage  $\theta$ , and percentage of inhibition efficiency increases with increasing ( $J_1$ - $J_3$ ) concentration and record the maximum value (94.00%  $J_1$ , 98.46%  $J_2$ , and 93.66% for  $J_3$ ) of inhibition efficiency at 3000 ppm concentration. The inhibition efficiency of the prepared additives

increases in the order  $J_2 > J_1 > J_3$ . The highest inhibition efficiency of inhibitor  $J_2$  may be attributed to the presence of two electron releasing groups (methyl group).

**Table 3:** <sup>1</sup>H-NMR data of the prepared (J) and (J<sub>1</sub>-J<sub>3</sub>) compounds

| Compound digestion | Group  | ppm  |
|--------------------|--|------|
| J                  | - CH <sub>3</sub>                                      | 0.96 |
|                    | - CH <sub>2</sub>                                      | 1.33 |
|                    | - (CH <sub>2</sub> ) <sub>n</sub>                      | 1.25 |
|                    | - CH <sub>2</sub> -CH-Br                               | 1.75 |
|                    | - CH-Br  | 3.83 |
|                    | - CO-CH <sub>2</sub>                                   | 1.68 |
|                    | - CH <sub>2</sub> -O                                   | 2.25 |
|                    | - O-CO-C   | 4.08 |
| J <sub>1</sub>     | - α, ε CH-Ph   | 6.43 |
|                    | - β, δ CH-Ph   | 7.04 |
|                    | - γ CH-Ph  | 6.58 |
|                    | - NH   | 4.00 |
|                    | - CO-O-CH <sub>2</sub>                                 | 4.08 |
|                    | - CO-O-CH <sub>2</sub> -CH <sub>2</sub>                | 1.57 |
|                    | - CO-CH <sub>2</sub>                                   | 2.25 |
|                    | - CO-CH <sub>2</sub> -CH <sub>2</sub>                  | 1.68 |
|                    | - CH-NH  | 2.84 |
|                    | - CH <sub>2</sub> -CH-NH                               | 1.48 |
|                    | - (CH <sub>2</sub> ) <sub>n</sub>                      | 1.29 |
|                    | - CH <sub>2</sub> -CH <sub>3</sub>                     | 1.33 |
|                    | - CH <sub>3</sub>                                      | 0.96 |
| J <sub>2</sub>     | - ε CH-Ph  | 6.19 |
|                    | - δ CH-Ph  | 6.65 |
|                    | - Ph-CH <sub>3</sub>                                   | 2.35 |
|                    | - β CH-Ph  | 6.64 |
|                    | - CO-O-CH <sub>2</sub>                                 | 4.08 |
|                    | - CO-CH <sub>2</sub>                                   | 2.25 |
|                    | - NH   | 4.00 |
|                    | - CH <sub>2</sub> -C-N                                 | 1.48 |
|                    | - (CH <sub>2</sub> ) <sub>n</sub> -CH <sub>2</sub> -CN | 2.84 |
|                    | - O-CH <sub>2</sub> -CH <sub>2</sub>                   | 1.57 |
|                    | - (CH <sub>2</sub> ) <sub>n</sub>                      | 1.29 |
|                    | - CH <sub>2</sub> -CH <sub>3</sub>                     | 1.33 |
|                    | - CH <sub>3</sub>                                      | 0.96 |
| J <sub>3</sub>     | - COOH   | 11   |
|                    | - α CH-Ph  | 7.91 |
|                    | - β CH-Ph  | 6.64 |
|                    | - NH   | 4.00 |
|                    | - CH <sub>2</sub> -CN                                  | 1.48 |
|                    | - CH <sub>2</sub> -CO                                  | 2.25 |
|                    | - CH <sub>2</sub> -O-CO                                | 4.08 |
|                    | - O-CH <sub>2</sub> -CH <sub>2</sub>                   | 1.57 |
|                    | - (CH <sub>2</sub> ) <sub>n</sub>                      | 1.29 |
|                    | - CH <sub>2</sub> -CH <sub>3</sub>                     | 1.33 |
|                    | - CH <sub>3</sub>                                      | 0.96 |

**Table 4:** Weight loss parameters of mild steel (CS-37) treated without and with the prepared corrosion inhibitors (J<sub>1</sub>-J<sub>3</sub>) at 308K

| Inhibitor      | Concentration "ppm" | Total weight loss (mg) | Corrosion Rate (mg.cm <sup>-2</sup> .h <sup>-1</sup> ) | Surface Coverage (θ) | IE %  |
|----------------|---------------------|------------------------|--|----------------------|-------|
| Blank          | 0                   | 650                    | 6.19   | .....                | ..... |
| J <sub>1</sub> | 250                 | 80                     | 0.7619   | 0.8769               | 87.69 |
|                | 500                 | 78.77                  | 0.7502   | 0.8788               | 87.88 |
|                | 1000                | 68                     | 0.6476   | 0.8953               | 89.53 |
|                | 2000                | 47.57                  | 0.4531   | 0.9268               | 92.68 |
|                | 3000                | 39                     | 0.3714   | 0.94                 | 94.00 |
| J <sub>2</sub> | 250                 | 59                     | 0.5651   | 0.9087               | 90.87 |
|                | 500                 | 41.20                  | 0.3924   | 0.9366               | 93.66 |
|                | 1000                | 26.712                 | 0.2544   | 0.9589               | 95.89 |
|                | 2000                | 15.20                  | 0.145  | 0.9766               | 97.66 |
|                | 3000                | 10.01                  | 0.0953   | 0.9846               | 98.46 |

|                |      |       |        |        |       |
|----------------|------|-------|--------|--------|-------|
| J <sub>3</sub> | 250  | 94.50 | 0.90   | 0.8546 | 85.46 |
|                | 500  | 84.76 | 0.8071 | 0.8696 | 86.96 |
|                | 1000 | 78    | 0.74   | 0.8800 | 88.00 |
|                | 2000 | 53.10 | 0.5057 | 0.9183 | 91.83 |
|                | 3000 | 41.21 | 0.3924 | 0.9366 | 93.66 |

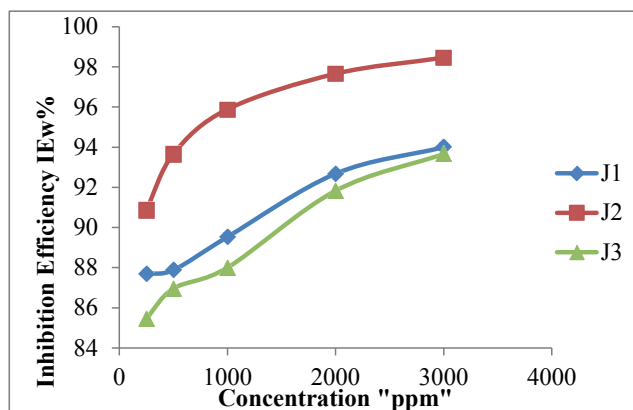


Figure 2: Variation of inhibition efficiency with different concentrations of the prepared inhibitors (J<sub>1</sub>-J<sub>3</sub>) in 0.5N HCl at 308K.

**Effect of temperature**

In order to calculate the activation energy (Ea) of the corrosion process and investigate the mechanism of inhibition, weight loss measurements were performed in the temperature range of 308-338K in absence and presence of 3000 ppm of the prepared inhibitors (J<sub>1</sub>-J<sub>3</sub>) at 0.5N HCl. It was found that the corrosion rate of mild steel CS-37 increases with increasing temperature. The results indicates that increasing temperature leads to a decrease of RT, hence increasing the corrosion rate of carbon steel as shown in **Figure 3**. A plot of ln corrosion rate (ln k) against the reciprocal of absolute temperature (1/T) was drawn graphically to obtain activation energy Ea, according to Arrhenius equation:

$$\ln K = -(Ea/RT) + \text{constant} \tag{5}$$

Where Ea equal to slope of this equation.

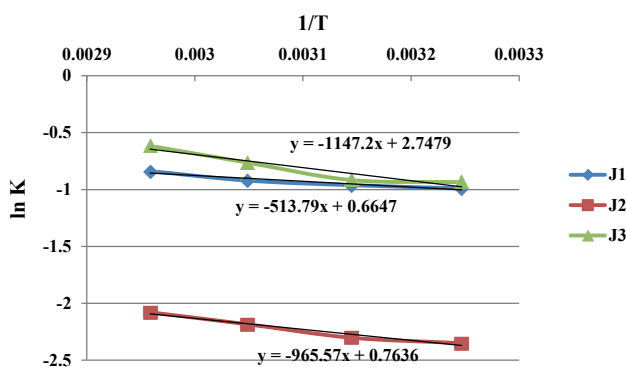


Figure 3: ln K vs. 1/T for the prepared corrosion inhibitors at (308-338)K.

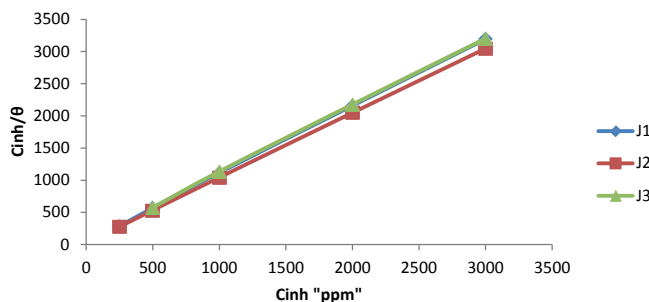
**Adsorption isotherm**

To understand the corrosion inhibition mechanism, the organic compound's adsorption behaviour on the carbon steel surface must be known. The plot of C<sub>inh</sub>/θ vs. C<sub>inh</sub>, **Figure 4** yielded a straight line, proved that the adsorption of the prepared corrosion inhibitors from the hydrochloric acid solution obeys Langmuir adsorption isotherm, which is presented by equation (6) [34].

$$C_{inh}/\theta = 1/K_{ads} + C_{inh} \tag{6}$$

Where C<sub>inh</sub> is the inhibitor concentration and K<sub>ads</sub> is the equilibrium constant for the adsorption/desorption process. From the intercepts of the straight lines on the C<sub>inh</sub>/θ-axis, one can calculate K<sub>ads</sub>, which is related to the standard free energy of adsorption, ΔG<sub>ads</sub>, as given by equation (7) [35]:

$$\Delta G_{\text{ads}} = -RT(\ln K_{\text{ads}} \times 55.5) \quad (7)$$



**Figure 4:** Langmuir isotherms for the adsorption of the prepared corrosion inhibitors ( $J_1$ - $J_3$ ) on carbon steel surface in 0.5N HCl at 308K.

The calculated free energy of adsorption ( $\Delta G_{\text{ads}}$ ) is given in **Table 5**. The negative values of  $\Delta G_{\text{ads}}$  indicated that the adsorption of the inhibitors on the metal surface is spontaneous. Generally, values of  $\Delta G_{\text{ads}}$  around  $20 \text{ kJ mol}^{-1}$  or lower are consistent with the electrostatic interaction between charged molecules and the charged metal surface (physisorption and chemisorption); It can be seen from **Table 5** that, calculated  $\Delta G_{\text{ads}}$  values indicated that the adsorption mechanism of the prepared corrosion inhibitors on carbon steel in 0.5N HCl solution is physical and chemical adsorption. The large values of  $\Delta G_{\text{ads}}$  and its negative sign are usually characteristic of strong interaction and a highly efficient adsorption. The aminated derivatives of soybean oil were generally chemisorbed at the metal surface and displace the adsorbed water and electrolytes from the surface. It is assumed that these N-containing functional groups act as electron pair donors to the electron depleted dehydrated metal surface. This interaction between the inhibitor and the metal surface is due to the formation of a bond between the N electron pair and the electron cloud at the metal surface.

**Table 5:** Adsorption parameters for the prepared corrosion inhibitors ( $J_1$ - $J_3$ ) on CS-37 in 0.5N HCl at 308K

| Inhibitor | $K_{\text{ads}}$ | $\Delta G_{\text{ads}}$ (KJ/mole) |
|-----------|------------------|-----------------------------------|
| $J_1$     | 42.272           | -19.549                           |
| $J_2$     | 29.684           | -18.659                           |
| $J_3$     | 51.043           | -20.024                           |

### Corroded metal concentration

The concentration of the corroded metal was calculated using chemical analysis method according to weight loss method at 308K. It was found that the concentration of the corroded metal decreases with increasing the concentration of corrosion inhibitors, while at 3000 ppm concentration of  $J_2$  corrosion inhibitor has the lowest concentration of corroded metal " $1.01 \times 10^{-5}$ " (**Table 6**).

**Table 6:** Concentration of corroded iron by chemical analysis according to weight loss method

| Inhibitor | Concentration "ppm" | Concentration of $[\text{Fe}^{+3}]$ Ion (mol/cm <sup>2</sup> .h) |
|-----------|---------------------|--|
| Blank     | 0                   | 0.0000726  |
| $J_1$     | 3000                | $6.65 \times 10^{-6}$  |
|           | 2000                | $8.11 \times 10^{-6}$  |
|           | 1000                | $1.159 \times 10^{-5}$   |
|           | 500                 | $1.343 \times 10^{-5}$   |
|           | 250                 | $1.36 \times 10^{-5}$  |
| $J_2$     | 3000                | $1.706 \times 10^{-6}$   |
|           | 2000                | $2.596 \times 10^{-6}$   |
|           | 1000                | $4.55 \times 10^{-6}$  |
|           | 500                 | $7.026 \times 10^{-6}$   |
|           | 250                 | $1.0119 \times 10^{-5}$  |
| $J_3$     | 3000                | $7.026 \times 10^{-6}$   |
|           | 2000                | $9.055 \times 10^{-6}$   |
|           | 1000                | $1.325 \times 10^{-5}$   |
|           | 500                 | $1.445 \times 10^{-5}$   |
|           | 250                 | $1.612 \times 10^{-5}$   |



## CONCLUSION

Three green corrosion inhibitors were prepared via reaction of brominated jojoba oil with different types of amines "aniline, 2,4- dimethyl aniline and 4-amino benzoic acid". The aminated derivatives were elucidated using FT-IR and <sup>1</sup>H-NMR, and they were evaluated as corrosion inhibitors for mild steel CS-37 at 0.5N HCl, using weight loss method and chemical analysis method at different temperatures "308-338"K. It was found that the inhibition efficiency "IE<sub>w</sub>%" increases with increasing the inhibitor concentration, while decrease with rising of temperature and the order of increasing the inhibition efficiency "IE<sub>w</sub>%" using the prepared corrosion inhibitors was as fellow: J<sub>3</sub><J<sub>1</sub><J<sub>2</sub>. The adsorption of the prepared inhibitors on metal surfaces from 0.5N HCl solution obeys Langmuir adsorption isotherm. The high value of adsorption equilibrium constant suggested that the prepared organic corrosion inhibitors were strongly chemically and physically adsorbed on the carbon steel surfaces. Chemical analysis method was used for the determination of concentration of the corroded iron according to weight loss calculations and it was found that the concentration of the corroded iron decreases with increasing concentration of the inhibitor, and the order of increasing the corroded iron was as fellow: J<sub>2</sub>>J<sub>1</sub>>J<sub>3</sub>.

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