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INHIBITION AND ADSORPTION EFFECTS OF *LAVANDULA* AND *RICINUS COMMUNIS* OILS ON MILD STEEL CORROSION IN H_2SO_4

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ABSTRACT

The inhibition and adsorption effects of Lavandula and Ricinus communis oils on mild steel corrosion in 0.5 M H_2SO_4 were studied at ambient temperature of 25°C. Gravimetric and potentiodynamic polarization measurement methods were used for the experiments. A Digi-Ivy potentiostat, interfaced with a computer for data acquisition and analysis was used for the potentiodynamic polarization experiments. The inhibition performance decreased with increasing concentration of the mixed inhibitors in the H_2SO_4 test medium. The low inhibitor concentrations of 2 ml, 4 ml, 6 ml/250 ml H_2SO_4 were relatively similar and had weight loss values of 0.152 g, 0.254 g, 0.21 g, respectively. At 336 h of the experiment, the concentration of 12 ml/250ml H_2SO_4 had the highest recorded weight loss value of 0.821g. The corresponding corrosion rate values for these concentrations were respectively 0.84 mm/year, 1.40 mm/year and 1.16 mm/year. The corrosion inhibition efficiency values for the 12 ml/250 ml H_2SO_4 inhibitor concentration was 96.35 % at 336 h. The experiment also achieved polarization resistance values of 3.90E+02 Ω; corrosion rate of 0.68 mm/year and current density of 5.84E-05 A cm^{-2} for the 2 ml/250 mL H_2SO_4 concentration test medium. Results of ba and bc indicated a mixed type inhibitor. Adsorption isotherm showed that the inhibitor protection mechanism followed both Frumkin and Freudlich models more than Langmuir isotherm model.

Keywords: corrosion, Lavandula oil, Ricinus communis oil, polarization, steel.

INTRODUCTION

Corrosion and protection of metallic materials, particularly the mild steel are of utmost concern to corrosion scientists and engineers with regard to their versatility in industrial use. Mild steel is generally low in cost, very widely available and has the properties that make it to be most versatile in metals' technology applications. However, its mild to severe susceptibility to corrosion in diverse environments such as in acidic, alkaline, aqueous and atmospheric, is disadvantageous. Mild steel must therefore be protected. Among the various means of the steel's protection is the use of inhibitors. Both inorganic and organic compounds had been effectively used for this purpose but they are not without their environmental unfriendliness. In very recent time, the use of plant extracts and oils for corrosion prevention and control (protection), particularly, as green inhibitors has been gaining some considerable research interest [1 - 14]. The reason for this is not unconnected with its environmental friendliness.

In general, corrosion inhibitors are chemical compounds whose reacting species interact with the metal surface and producing a film barrier at the metal-environment interface to inhibit corrosion reactions on the metal's surface. In this instance, the mechanism of the corrosion inhibition could be either by altering the anodic or cathodic polarization behaviour, increasing the electrical protection of the metal's surface, reducing the spreading of ions to the metal surface or a combination of any of the three with another one. The influence of inhibitors is often associated with physical or chemical adsorption. This phenomenon had been related to the

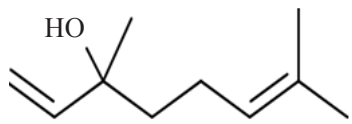
presence of hetero atoms (N, O, S, and multiple bonds or aromatic rings in the inhibitor [15]. These atoms could be seen more in organic inhibitors.

In this work, two different oil types were used as a corrosion green inhibitor. They are: Lavender oil (*Lavandula latifolia*) and the Castor oil (*Ricinus communis L*). Lavender oil is an essential oil obtained by distillation from the flower spikes of certain species of lavender. The oil is a complex mixture of phytochemicals, including linalool and linalyl acetate. The main chemical components of lavender oil are: α-pinene, limonene, 1, 8-cineole, cis-ocimene, trans-ocimene, 3-octanone, camphor, linalool, linalyl acetate, caryophyllene, terpinen-4-ol and lavendulyl acetate. The second oil used was the castor oil. The unique structure of castor oil is known to offer some interesting properties that make it appropriate for various industrial applications. The main principal constituents of castor oil are: up to 90 % ricinoleic (12-hydroxy-9-cis-octadecenoic acid), 4 % linoleic, 3 % oleic, 1 % stearic and less than 1 % linoleic fatty acids. The presence of the hydroxyl group in ricinoleic acid (RA) and its derivatives provide a functional group location for performing a variety of chemical reactions that include halogenation, dehydration, and alkoxylation [16]. The chemical structures of the two oils used are presented below. This investigation aims at studying the inhibition effect of *Ricinus communis* oil and *Lavendul*a oil on the corrosion resistance of mild steel in dilute sulphuric acid medium. The results obtained are expected to be of scientific, technological and economic benefits.

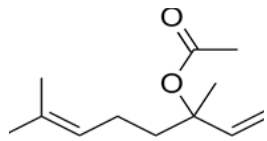
EXPERIMENTAL

Materials

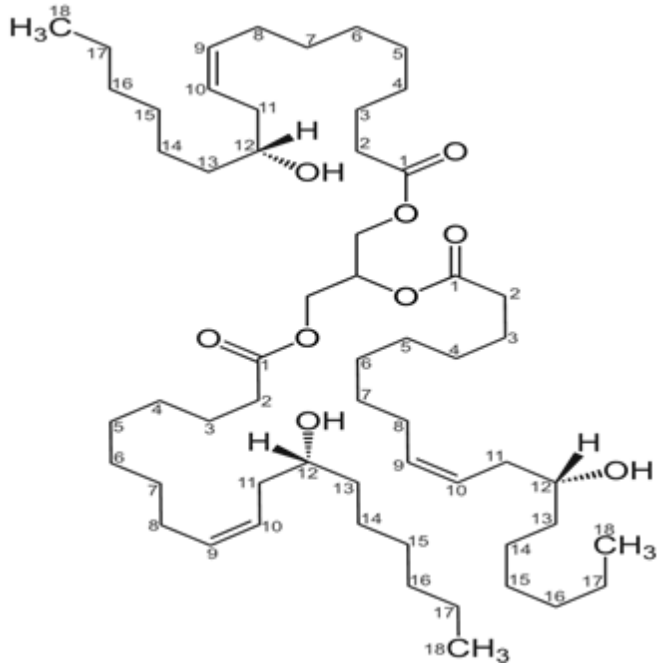
The cylindrical mild steel sample of 12 mm diameter was each cut into average size of 12 mm x 10 mm coupons for weight loss measurements and also for potentiodynamic polarization measurements. Test samples used for the weight loss experiment were de-scaled with a wire brush,



Linaloo (Lavender oil-Terpenes/Monoterpenols)



Linalyl acetate (Lavender oil-Terpenes Ester) [17]



Triester of glycerol and Ricinoleic acid (Castor oil) [18]

ground with various grades of emery paper and then polished to 6 μm,. They were further rinsed in distilled water to remove any corrosion products and then cleaned with acetone to degrease. The samples were fully immersed in 0.5 M H_2SO_4 acidic medium. Another set of samples for the corrosion polarization experiments were cleaned in the same manner as those for the weight loss experiment. They were subsequently mounted in resin to ensure that only the tested surface of the sample was exposed to the corrosive medium. Before mounting, copper wire was spot-welded to each of the samples. The surface of the samples were thoroughly cleaned and prepared for experimental use with silicon carbide papers of up to 1000 grade before being cleansed in distilled water and dried with acetone.

Experimental setup

The experiments were set up in 6 different environments, one control experiment and five other experiments with different concentrations of the castor and lavender oil inhibitors in 0.5 M H_2SO_4. The concentrations of the oil inhibitors used were: 0, 2, 3, 4, 6, 8, 10 and 12 g /200 ml H_2SO_4. The test media without added inhibitor served as the control experiment.

Weight loss experiments

Weighed test species were fully and separately immersed in 200 ml of the test environment at specific concentrations of the inhibitors for 336 hours at room temperature. The method involves exposing a specimen of material to a process environment over a period of time, taking the weight of the sample at a time interval. Each of the test samples was taken out every 24 hours, washed with distilled water, rinsed with acetone, dried and re-weighed. Plots of weight loss (g), corrosion rate (mm/year) and percentage inhibition efficiency (% IE) versus exposure time in hours were made from the readings recorded in the tables.

From the weight loss data, corrosion rate (C_r) values were calculated from the formula:

Corrosion Rate (in mm/year) =

$$\frac{87.6 \times \text{Weight Loss(g)} \times 1000\ \text{mg}}{\text{Metal Density (g/cm}^3) \times \text{Surface Area (cm}^2) \times \text{Exposure Time (hours)}}$$

The per cent inhibition efficiency (% IE) is calculated as:

I.E % = [(Wl– W2)/W1] * 100 (1)

Or: (1-W2/W1)*100 (2)

where W1 is the weight loss of the control sample and W2 is the weight loss of the other samples respectively.

Potentiodynamic polarisation experiments

The electrochemical polarisation techniques were performed on the prepared test specimens immersed in the acid test environments with and without the addition of various inhibitor concentrations. Tafel plots were generated in this experiment by polarizing the specimen about 300 mV in the anodic direction (positive going potential) and cathodically (negative going potential) from the corrosion potential, Ecorr. The resulting current is plotted on a logarithmic scale. The data generated include current density, corrosion rate, and polarisation resistance. These will be described and discussed below in more details.

Optical microscopy/micrographs

Photomicrographs were made of the relevant and selected surfaces of the test samples after use for weight loss experiments.

RESULTS AND DISCUSSION

Weight loss method

The results of the weight loss performed on the corrosion inhibition of mild steel are shown in Fig. 1 as the curve of weight loss versus time for a period of 336 hours. It could be observed from the Fig. 1 that the weight loss value of 4.162 g at 336 hours of the experiment of the control sample was much greater than those of the other test electrodes with various concentrations of the inhibitor addition. The weight loss values of other test electrodes with relatively low inhibitor concentrations addition of 2 ml, 4 ml, and 6ml/200 ml H_2SO_4 were relatively similar and had weight loss values of 0.152 g, 0.254 g, 0.21 g, respectively. The 12 ml/200 ml H_2SO_4 concentration (the highest concentration used) had the highest weight loss value of 0.821 g at 336 hours. In this experiment, the lowest concentration of the mixed inhibitors was the most effective.

The corrosion rate results of the weight loss experiments performed for the corrosion inhibition of mild steel in 0.5 M H_2SO_4 for 336 hours are presented in Fig. 2. It could be observed here that the corrosion rate of the test sample without inhibitor concentration is very much greater than those with added inhibitor concentrations. This achieved a corrosion rate value of 22.92 mm/year. The corrosion rates for the test samples with added

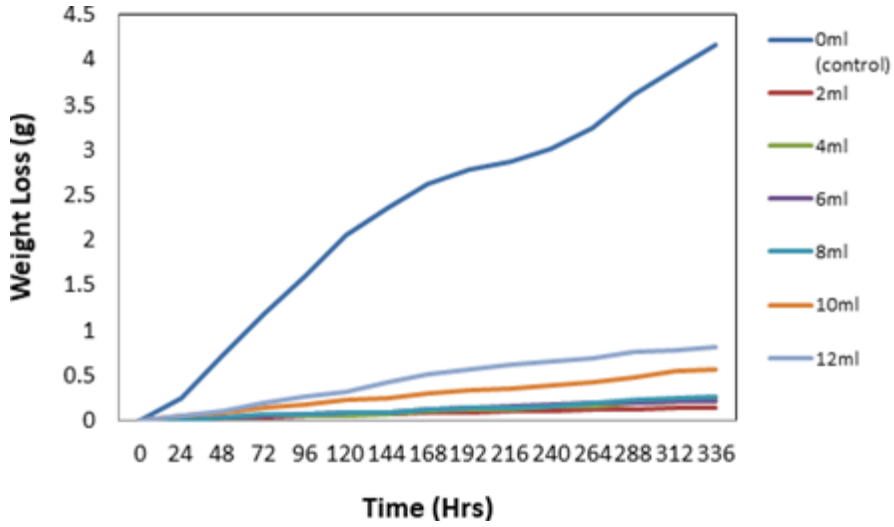


Fig. 1. Weight loss versus exposure time for mild steel immersed in 0.5 M H_2SO_4 with different concentrations of added mixed lavender/castor oils (LACA) as inhibitors.

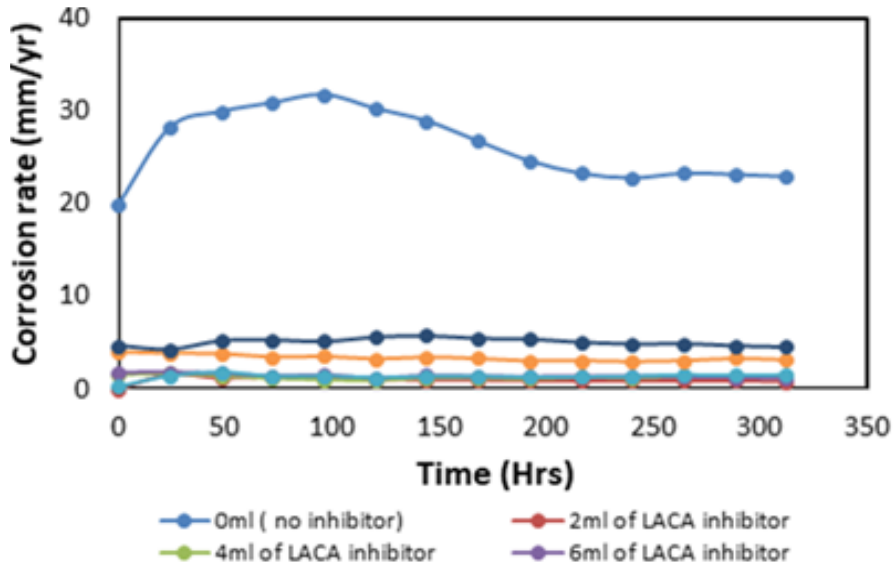


Fig. 2. Corrosion rate versus the exposure time for mild steel immersed in 0.5 M H_2SO_4 + different concentrations of mixed lavender/castor oils (LACA) inhibitor addition.

inhibitor concentrations of 2 ml, 4 ml, and 6 ml/200 ml H_2SO_4 had corrosion rate values of 0.84, 1.40 and 1.16 mm/year, respectively.

It is apparent in this experiment that the test electrode with the lowest concentration of LACA inhibitor addition gave the most effective corrosion inhibition in the sulphuric acid test medium. Likewise, the test with the highest inhibitor concentration had the least inhibitive effect.

The inhibition efficiency (IE) results of the weight loss experiments performed for the corrosion inhibition of mild steel in sulphuric acid test medium for 336 hours are presented in Fig. 3. It could be observed that the inhibition efficiency of the sample with the highest concentration was much lower than those of the other samples which contained lower concentrations, achieving % IE value of 80.27 at 336 hours. The inhibition efficiency values of samples with inhibitor concentrations of 2 ml, 4 ml, and 6 ml/200 ml H_2SO_4 remained generally constant from the onset and had the inhibition efficiency values of 96.35 %, 93.90 % and 94.95 %, respectively. The sample with the lowest concentration of LACA inhibitor addition gave the most effective corrosion inhibition in the sulphuric acid test medium. The

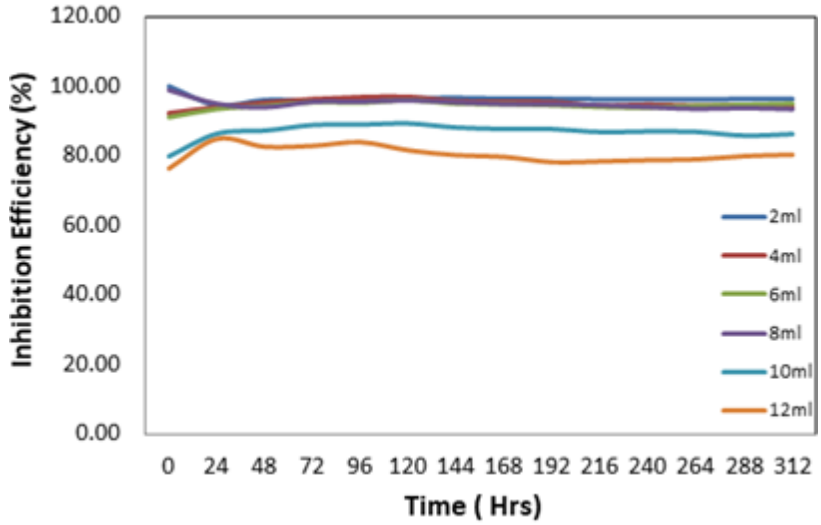
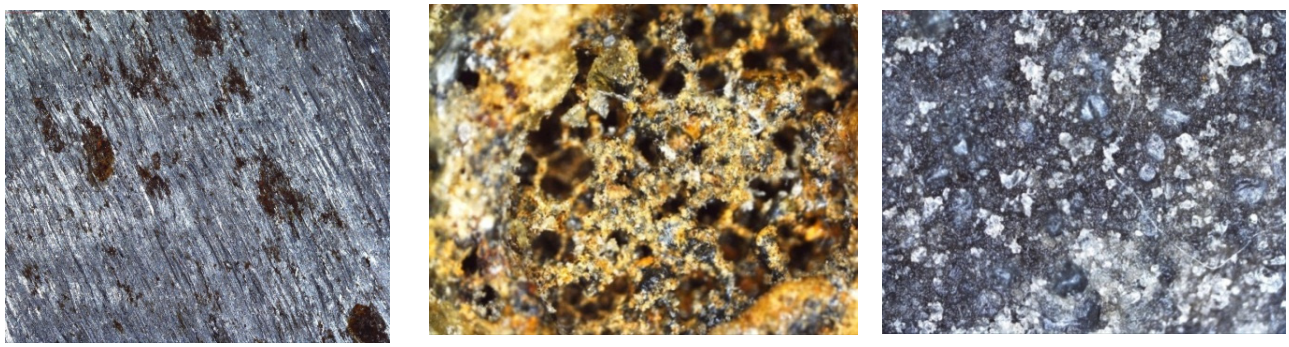


Fig. 3. Inhibition efficiency of mild steel immersed in 0.5 M H_2SO_4 + different concentrations of mixed lavender/castor oils (LACA) inhibitor.



A B C

Fig. 4. Surface microstructure of uninhibited and inhibited steel surface in the acidic media: A) before immersion; B) immersion in H_2SO_4 without inhibitor; C) immersion with 12 ml/200 ml H_2SO_4 LACA inhibitor concentration.

test electrode with the highest concentration of LACA inhibitor was the least effective.

Optical microscopic results

Photomicrographs of the samples were taken at the end of the weight-loss experiment. Representative copies are shown in Fig. 4. The sample B without added inhibitor was visibly very much corroded with a combination of both general and pitting corrosion. The surface feature of sample C with 12 ml/200 ml H_2SO_4 LACA inhibitor concentration addition showed very little moderate corrosion effect. The surface microstructural features here are in agreement with the gravimetric results obtained above. Thus the oils in combination could be observed to be synergistically inhibitive in the test environment on the mild steel corrosion.

Adsorption Isotherm

Molecular adsorption to the metal electrode's surface could be used to explain the phenomena of corrosion inhibition in this investigation. A number of factors that include: the nature and surface charge of metal, the type of aggressive media, the distribution of charge in molecule, and the chemical structures of organic compounds can influence the molecular adsorption process [19]. The relation below has been used to determine the value of the adsorption equilibrium constant, k, and the standard free energy of adsorption [20]:

$$k = \frac{1}{55.5}\exp\left(-\frac{\Delta G^o_{ads}}{RT}\right)$$

where ΔG^o_{ads} is the standard free energy of adsorption; R is the molar gas constant and T is the absolute

Table 1. Adsorption isotherm data for mild steel in 0.5 M of H_2SO_4 at 336 hours.

MCS Samples	Weight Loss (g)	LACA Concentration (%)	LACA Concentration (Molarity)	Corrosion Rate (mm/yr)	LACA Inhibition Efficiency (%)	Surface Coverage (θ)	θ/1-θ
A	4.162	0	0	24.49	0	0	0
B	0.152	1.0	1.07E-02	24.51	0.12	0.0012	0.0012
C	0.254	1.5	1.60E-02	20.33	16.99	0.1699	0.2047
D	0.210	2.0	2.13E-02	18.07	26.19	0.2619	0.3549
E	0.275	2.5	2.67E-02	13.47	44.98	0.4498	0.8176
F	0.573	3.0	3.20E-02	14.26	41.77	0.4177	0.7173
G	0.821	3.5	3.74E-02	4.93	79.86	0.7986	3.9643

temperature.

The negative values of ΔG^o_{ads} obtained indicates the spontaneous adsorption process and the stability of the adsorbed inhibitor layer on the metal surface.

In this investigation, three different types of adsorption isotherm models were used: (i) Langmuir isotherm; (ii) Frumkin isotherm; (iii) Freundlich isotherm (Figs. 5 - 7). The values of the standard free energy of adsorption for castor bark powder had been determined to be -16.92 kJ /mol [21]. This indicates the physiosorption mode of adsorption. Values of ΔG^o_{ads} that are -20 kJ/mol and above, i.e. less negative, have been associated with

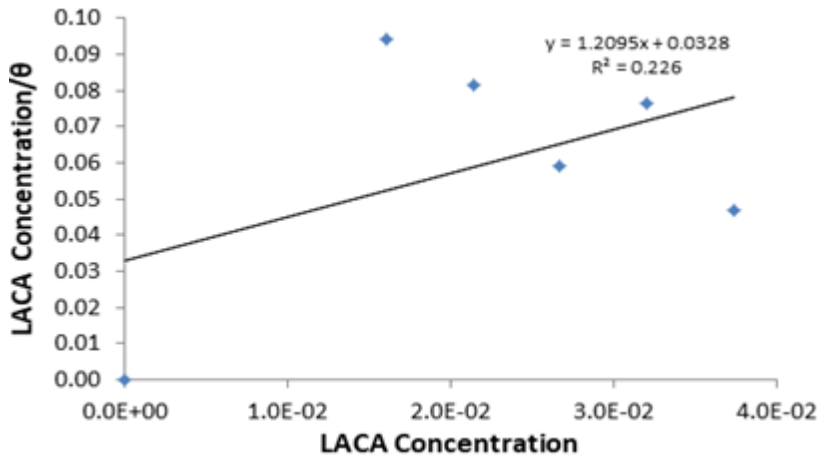


Fig. 5. Langmuir isotherm plot for the adsorption of Lavender/Castor oils (LACA) on mild steel in 0.5 M H_2SO_4.

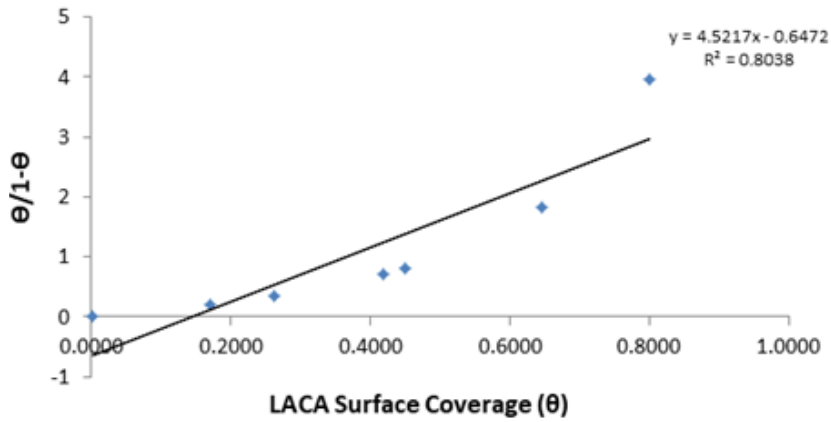


Fig. 6. Frumkin isotherm plot for LACA adsorption on mild steel in 0.5 M H_2SO_4.

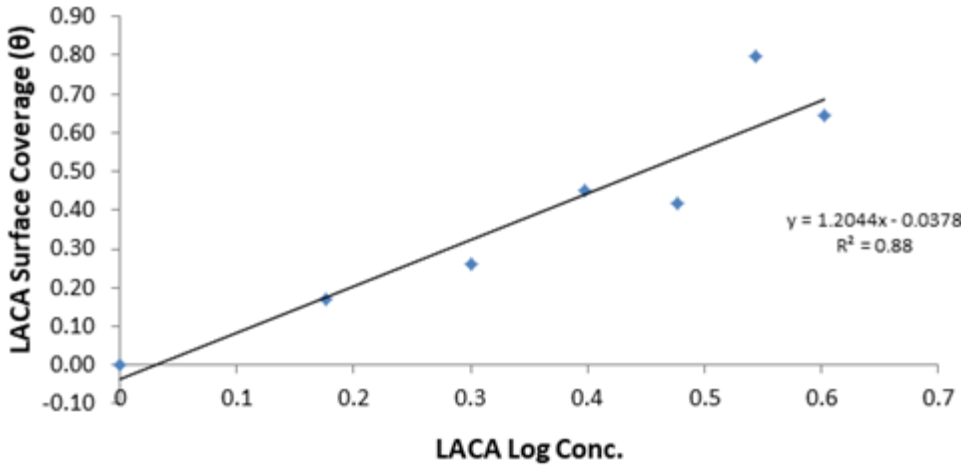


Fig. 7. Freundlich isotherm plot for LACA adsorption on mild steel in 0.5M H_2SO_4.

physical adsorption (physiosorption) which involves electrostatic interaction between charged atoms and the charged metal, while those around -40 kJ/mol and more negative are generally associated with chemical adsorption. Frumkin and Freundlich adsorption isotherms give the best fit. Freundlich adsorption isotherm is given by the relations [22]

$$\theta = KC^n$$

$$Log\ \theta = n\ Log\ C + \ln K$$

where θ is the degree of surface coverage; *K* and *n* are coefficients; and *C* is the inhibitor concentration. The adsorption isotherm showed that the inhibitor protection mechanism followed both the Frumkin and the Freundlich models more than the Langmuir isotherm model.

Potentiodynamic polarisation

The potentiodynamic polarization curves for mild steel immersed in 0.5 M of sulphuric acid with different mixed Lavandula and Ricinus communis oils inhibitor concentrations and without inhibitor addition are presented in Fig. 8. The parametric results of the polarization experiments are shown in Table 2. It can be observed that with the control sample, (the solution without inhibitor) the corrosion rate was 8.41mm/year, corrosion current density was 6.18E-04 A/cm^2 and a polarization resistance value was 4.16E+01 Ω. With the lowest inhibitor concentration of 2 ml/200ml 0.5 M H_2SO_4 the corrosion rate has a value of 0.68 mm/year, a corrosion current density value of 5.84E-05 A/cm^2 and a polarization resistance value of 3.90E+02 Ω. The value

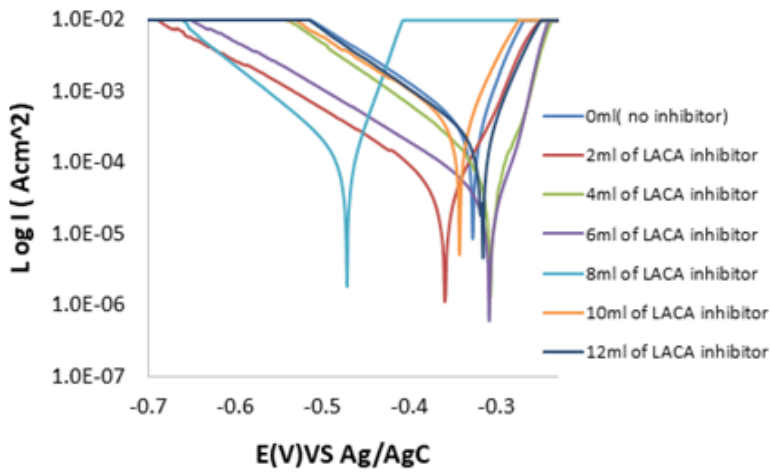


Fig. 8. Corrosion polarization of mild steel in H_2SO_4 with and without added inhibitor.

Table 2. Corrosion Polarization Results for the H_2SO_4 medium.

Sample	LACA Conc. (%)	MS Corrosion Rate (mm/y)	Corrosion Current (A)	Corrosion Current Density (A/cm^2)	Corrosion Potential (V)	Polarization Resistance, Rp (Ω)	Cathodic Tafel Slope, Bc (V/dec)	Anodic Tafel Slope, Ba (V/dec)
A	0	8.41	6.18E-04	6.18E-04	-0.328	4.16E+01	-8.54E+00	-4.117E-16
B	2.0	0.68	6.60E-05	5.84E-05	-0.36	3.90E+02	-7.84E+00	2.95E+01
C	4	1.12	1.09E-04	9.62E-05	-0.308	2.36E+02	-9.26E+00	8.85E+00
D	6.0	0.60	5.85E-05	5.18E-05	-0.309	4.39E+02	-7.84E+00	5.92E+00
E	8	2.60	2.53E-04	2.24E-04	-0.322	1.02E+02	-9.89E+00	1.03E+00
F	10.0	6.41	6.25E-04	5.53E-04	-0.343	4.11E+01	-6.83E+00	3.03E+00
G	12	4.45	4.34E-04	3.84E-04	-0.316	5.92E+01	-8.61E+00	2.07E+00

of corrosion rate continued to decrease with increasing inhibitor concentration up until sample F with the concentration 10 ml/200 ml 0.5 M H_2SO_4 and 6.41 mm/year corrosion rate; its current density was 5.53E-04 A/cm^2 and with a polarization resistance value of 4.11E+01 Ω. Though comparatively lower than the former, this trend of increased corrosion rate was also shown with the highest inhibitor concentration value of 12 ml/200 ml of 0.5 M H_2SO_4 that was used. The results here were in agreement with those of gravimetric method. It could be plausibly expressed from the results parameters that the best effective inhibition values were achieved with the acid inhibitor concentrations of 2, 4, and 6 ml/200 ml of 0.5 M H_2SO_4 as indicated by the corrosion rate, current density and polarization values.

The mixture of lavender and castor oils have been found in this investigation to be synergistically effective as green inhibitors at different concentrations in the sulphuric acid test medium as observed in the results above. Castor oil had been used separately as an inhibitor [21]. Also reported was the use of lavender as a green inhibitor [24]. These two oils have different complex chemical compositions that may exhibit electrochemical activity of inhibiting active corrosion reactions processes. The presence of heteroatoms in their chemical structure could account for their inhibitive action in combination. These molecules of these atoms could adsorb to the reaction sites on the metal surface providing a stable film that hinders the metal/environment (acid) interfacial reactions [25]. It is obvious that the results of gravimetric and potentiodynamic polarization experiments and surface microstructural examinations are in agreement on the inhibitory property of these combined green inhibitors in the sulphuric acid test medium.

CONCLUSIONS

1. A good inhibitory performance was established when the combined *Lavandula* snd *Ricinus communis* oils are used at lower concentrations of 2, 4, and 6 ml/200 ml H_2SO_4.

2. Results of gravimetric experiments, inhibitor efficiency and potentiodynamic polarization are in agreement regarding the inhibition effectiveness of the green inhibitors.

3. Adsorption isotherm showed that the inhibitor protection mechanism followed both Frumkin and Freundlich models better than Langmuir isotherm model. Results of ba and bc indicated a mixed type inhibitor.

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