ISSN(Online) : 2319 - 8753
ISSN (Print) : 2347 - 6710

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 4, Issue 3, March 2015

Evaluating the Behavior of Aluminum Corrosion in Hydrochloric Acid in the Presence Aqueous Extract of Olive Seeds

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ABSTRACT: Corrosion of aluminum in HCl solutions and its inhibition by aqueous extract of Olive seeds (OS) extract have been reported. The study was carried out using weight loss, hydrogen evolution at 30 and 40°C and the results were supporting by surface examination study (SES) and electrochemical impedance spectroscopy (EIS) measurements at 30°C. Experimental results indicated that the corrosion of aluminum increased with increasing acid concentration and rising temperature. The presence of OS extract inhibit the general and pitting corrosion of aluminum in HCl solutions. This effect was remarkably increased by increasing the concentration of OS extract. The inhibition efficiency increased with rising temperature in the presence of inhibitor, especially at low concentrations. The adsorption of the inhibitor was also found to be spontaneous, chemical and consistent with the assumptions of Langmuir adsorption isotherm.

KEYWORDS: Aluminum corrosion, Corrosion inhibition, Olive seeds, hydrochloric acid solutions.

I. INTRODUCTION

Aluminum is an important metal in industry owing to its many excellent characteristics including good electrical and thermal conductivities, low density, high ductility, and good corrosion resistance. It is widely used as a material for automobiles, aviation, household appliances, containers, and electronic devices, which get corroded easily in the presence of acids[1,2] such as hydrochloric (HCl), sulfuric (H_2SO_4) and phosphoric (H_3PO_4) acid, ect.

Hydrochloric acid is one of the most widely used agents in the industrial sector. Due to the aggressiveness of acid solution to aluminum, the use of inhibitor to prevent the metal dissolution process will be inevitable[3].
The protection of aluminum from the corrosive chloride attack has been studied by many investigators using organic and inorganic compounds as corrosion inhibitors[1,2,4-12]. It has been reported[13] that the inhibitor molecules are bonded to the metal surface by chemisorption, physisorption, or complexation with the polar groups acting as the reactive centers in the molecules.

Most synthetic inhibitors are highly toxic and thus lead to the investigations on the use of a naturally occurring corrosion inhibitor which at the same time is not harmful to both human and the environment[14].

Olive stone and seeds are an important product generated in the olive oil extraction and pitted table olive industries. As a lignocelluloses material, the hemicelluloses, cellulose, fatty acids, vitamins, lignin and other water soluble components are the main components of olive seeds as wells as protein, fat, phenols, free sugars composition. Its primary fatty acids are oleic and linolenic acids. Oleic acid is monounsaturated and makes up about 55%-85% while linoleic acid is polyunsaturated and makes up about 9% of the oil. The main use of this biomass is as combustion to produce electric energy or heat. Other uses such as activated carbon, furfural production, plastic filled, abrasive and cosmetic or other potential uses such as biosorbent, animal feed or resin formation have been cited[15].

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The objective of the present work is to report the evaluating of behavior of aluminum corrosion and the effect of aqueous extract of Olive seeds as anticorrosion for aluminum in 1M hydrochloric solution at different temperatures using chemical (ML& HE), electrochemical (EIS) and surface examination study (SES).

II. MATERIALS AND METHOD

The corrosion behavior of *OS* extract on aluminum was determined via gas-volumetric (hydrogen evolution (HE)) and gravimetric (mass loss (ML)) measurements. The adsorption nature and surface morphology analysis using scanning electron microscope (SEM) were also determined.

1- Preparation of Olive seeds extract

Olive seeds were collected and washed under the running tap water and then were dried for three days to remove water before being ground into fine powder. Initially, the dried powder was diluted with deionized water in the ratio of 1:10(w/v). The solution was then placed in a hot water for 24h and then filtered to obtain the stock solution.

2-Specimen preparation

Aluminum specimen having the composition (wt%) of 0.08 C,0.01 Si, 1.26 Mn, 0.02 P and remaining Al was used. The sample with 5x1 cm^2 dimensions was used.

3- Solutions preparation

About 1 M HCl solution was prepared by the dilution of 37% HCl using deionized water. The concentration range of *Olive seeds* extract employed was varied from 1 to 10%v/v.

4- Gravimetric (ML) measurements

The aluminum specimen was immersed in different concentrations of HCl. For inhibition study, the mass loss of aluminum specimen was determined after 1h of immersion in presence of different concentrations of *Olive seeds* extract in 1M HCl at 30°C and 40°C. Then the specimen was washed, dried and weighed. The percentage inhibition efficiency by Gravimetric (ML) measurements ($Inh._{ML}\%$) was calculated using the following formula:

$$Inh._{ML}\% = W_0 - W_i / W_0 \times 100 \quad (1)$$

where W_0 and W_i are the weight loss values in the absence and in the presence of the inhibitor.

5- Gas-volumetric (HE) measurements

The procedure adopted for this technique was as previously reported[5,6,15,16]. The progress of corrosion reaction was monitored by careful measurement of the volume of hydrogen gas evolved at fixed time intervals. The inhibition efficiency by Gas-volumetric (HE) measurements ($\%Inh._{HE}$) was obtained from:

$$(Inh._{HE})\% = (1 - V^1_{Ht} / V°_{Ht}) \times 100 \quad (2)$$

where V^1_{Ht} and $V°_{Ht}$ are the volume of hydrogen evolved at time t for Al corrosion in the presence and absence of inhibitor, respectively.

6-. Surface Examination Study (SES)

The nature of the film formed on the surface of metal specimen was analyzed by SES. For taking photograph images, the aluminum specimen was immersed in the solution without inhibitor as well as in the solution containing optimum concentration of inhibitor (3 and 10%v/v) separately for 60 minutes. Then, the specimen was removed and rinsed with rectified spirit quickly. The images were taken using Digi scope II V2.

7- Electrochemical Impedance Spectroscopic (EIS) Study

EIS measurements were carried out over a frequency range of 0.5 kHz to 10000 Hz, with a signal amplitude perturbation of 10 mV at open circuit potential using an impedance spectrum analyzer (ACM Gill AC) connected with a computer. Nyquist plots were determined after 1h of immersion in the presence of 3%v/v and 10%v/v of *Olive seeds* extract in 1M HCl at 30°C.
efficiency ($Inh.C_{dl}\%$) of the investigated inhibitor was calculated from the following equation:

$$(Inh._{cdl})\% = [(1 - C_{dl}) / C_{dl°}] \times 100 \quad (3)$$

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III. RESULTS AND DISCUSSION

1-EFFECT OF HCL CONCENTRATION

The corrosion rates of aluminum in different concentrations of HCl solution (0.25-1.5M) was discussed by monitoring the weight loss and the volume of hydrogen gas evolved at fixed time. Figure 1 shows representative plots of the volume of hydrogen gas evolved as a function of time in different concentrations of HCl solution at 30^o C. Table 1 shows the corrosion rates obtained from the slope of the linear part of the gasometry plots for the test solutions and corrosion rates obtained from the mass loss technique. The corrosion is attributed to the presence of water, air and H^+, which accelerate the corrosion process. It is clear that the corrosion of the aluminum in HCl increases with the concentration of the acid. This observation is attributed to the fact that the rate of chemical reaction increases with increasing acid concentration.

1-EFFECT OF TEMPERATURE ON THE CORROSION OF ALUMINUM

There is a progressive increase in weight loss of aluminum and hydrogen evolved in 1M HCl as the temperature is increased from 30°C to 60°C as shown in Fig. 2. This signifies that the dissolution of the metal increased at higher temperatures. This observation is attributed to the general rule guiding the rate of chemical reaction, which reveal that chemical reaction increases with increasing temperatures. Also, increased temperature favors the formation of activated molecules, which may be doubled in number, with 10ºC rise in temperature, thereby increasing the reaction rate. This is because the reactant molecules gain more energy and are able to overcome the energy barrier more rapidly [17,18]. An increase in temperature may also increase the solubility of the protective films on the metals, thus increasing the susceptibility of the metal to corrosion[19,20]. The solubility of oxygen gas decreases with increase in temperature. Thus oxygen concentration is expected to be more at higher temperature which in this case is higher at 60°C than at 50°C, 40°C and 30°C. The presence of high concentration of oxygen thereby causes the metal to corrode faster. Also the protective film which is solid becomes more soluble as the temperature is increased.

2-INHIBITION ACTION OF OLIVE SEEDS EXTRACT ON THE CORROSION OF ALUMINUM

Figures 3&4 represent plots of hydrogen gas evolved for aluminum corrosion in the presence of different concentrations of inhibitor (*OS*) at 30^{o}C and 40^{o}C, respectively. It is observed from the figures that the volume of hydrogen gas evolved varies linearly with time and the slope of the liners were less in the presence of *OS* extract. The general decrease in hydrogen gas evolution with time as concentration of additives increased from 1%v/v to 10%v/v confirms that the presence of the additives in 1M HCl solution reduced the corrosion of aluminum. The hydrogen evolution technique enables us to assess the inhibitory effect of the inhibitor at 1M HCl corrodent concentrations, this means that the corrosion rate of aluminum was decreased with increasing the concentration of *OS* extract compared to the free acid solution at 30°C & 40°C.

Calculated values of the corrosion rate and inhibition efficiency (*Inh. %*) for the different systems from HE and ML in the presence of *OS* extract compared to the blank solution are presented in Tables 1-4. The data presented in the Tables follow the same trend observed for two methods HE and ML. Inhibition efficiency is also observed to generally increase with rising temperature, suggesting that metal/inhibitor interactions become high at low concentrations of *OS* extract (1,3 and 5%v/v), while at 10%v/v, the *Inh.%* at 40°C is lower than that at 30°C. This means that at low concentrations of *OS*, chemical adsorption will be occur. At high concentrations of *OS* the adsorption is physical.

3-SCANNING ELECTRON STUDY (SES)

The surface morphology of aluminum surface after 1hour immersion in the test solution at 30°C in the absence and presence of lower and higher concentration of *OS* has been done as in Figs.5 (a,b,c). Figure 5(a) illustrates the effect of 1M HCl on the aluminum surface, it appears the general corrosion and pitting corrosion. The pits are deep and contain a local cell. Pitting corrosion may increase the corrosion rate.

In Fig .5(b),the presence of thin porous and protective layer on aluminum surface contain of numerous pits less than that appears in case of HCl alone was observed in presence of 3% v/v of *OS* extract in 1M HCl. It obvious that $Inh._{ML}$% is about 46.94% in this case. On comparing Fig. 5 (c), which illustrates the effect of 10% v/v of *OS* extract, $Inh._{ML}$% is about 97.56. It appears that the disappearance of pits and the formation of an adsorbed film on aluminum surface due to adsorption of *Olive Seeds* components lead to high corrosion decrease at this concentration. This confirms the results obtained from mass loss and hydrogen evolution studies.

4-ELECTROCHEMICAL IMPEDANCE SPECTROSCOPIC (EIS) STUDIES

The corrosion behavior of aluminum in 1 M HCl in the presence of 3%v/v and 10%v/v was investigated by the EIS method at 30°C after immersion for 1 h. The Nyquist plots of aluminum in 1 M HCl in absence and presence of *OS*, 10 & 3%v/v are shown in Fig.6. The Nyquist plots are significantly changed on addition of inhibitor. The impedance of the inhibited system increased with inhibitor concentration. The results were recorded in Table 5, and it clear that this result is supports the previous results.

4- ADSORPTION BEHAVIOR OF OS ON ALUMINUM SURFACE IN HCL ACID SOLUTION

The adsorption process depends on the structure of the inhibitor (extract molecules), the nature and charge of metal surface, temperature, the type of electrolyte solution and the number of active centres on the surface. The adsorption process can be described by two main types of interactions: physical adsorption and chemical adsorption, or both[21,22].

The decrease in corrosion rate of aluminum sample by adding the *Olive Seeds* may be due to the adsorption of components of the extract on the aluminum surface or to form a protective barrier layer which separate the aluminum surface from the center of corrosion[23,24].

Figures (7&8) illustrate the relationship between *Inh.%* deduced from different methods and the logarithm of the concentration of *OS* extract at both 30°C and 40°C, respectively. Figure 7 shows an adsorption curves with *S*-shape adsorption isotherm at 30°C, which indicates that the interaction takes place in one step attributed to the formation of a single layer of extract molecules adsorbed on aluminum surface. At 40°C the curve is extremely *S*-shape but not perfect, Fig. 8.

The plots of C/Θ versus C, inhibitor concentration (Langmuir isotherm [25-27]) using equations 3, yielded straight lines for *OS* extract and with slopes close to 1 (Figs. 9&10) at 30°C and 40°C, respectively.

$$C/\Theta = 1/K_{ads} + C_{inh.} \qquad (4)$$

where C represents the concentration of extract, Θ degree of surface coverage and K_{ads} equilibrium constant of adsorption.

This results indicate that the adsorption of compounds in *OS* extract under consideration on aluminum/acidic solution interface follows the Langmuir adsorption isotherm. Table 6 represents the results derived from the application of Langmuir relationship C/Θ Vs. $C_{inh.}$, where the adsorption parameters are recorded for the studied extracts (*OS)* from different techniques. The results applied give linear correlation coefficients (r^2) close to one which confirms that the adsorption of the extract components on the surface of aluminum sample under study is the basic step in the process of inhibition.

The activation energy was calculated using the Arrhenius equation (5) below:

$$E_a = (2.303RT_1T_2\log R_1/R_2)/1000(T_2/T_1) \qquad (5)$$

Where E_a= activation energy, R = is the gas constant in Joule (J), T= Absolute temperature in Kelvin , R_1 and R_2 are rate constants at T_1 (303K) and T_2 (313K), respectively.

The average activation energy for the dissolution of aluminum in HCl solution between 30 and 40°C in the presence of *OS* extract was found to be 78.30 and 96.29 kJmol^{-1} from ML and HE, respectively. Two types of adsorption processes had been distinguished [28-31], physisorption in which the activation energy is less than about 40 kJmol^{-1} and chemisorption where the activation energy is greater than 80 kJmol^{-1}. On the basis of the experimentally determined activation energy values, the additive is chemically adsorbed on the aluminum sample. Therefore, it is probable that a monolayer protective coverage on the entire aluminum surface was obtained.

The increase in inhibition efficiency as temperature rises support the fact that the additive is chemsically adsorbed on the aluminum surface.

ISSN(Online) : 2319 - 8753
ISSN (Print) : 2347 - 6710

International Journal of Innovative Research in Science, Engineering and Technology

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By examining the chemical composition of *Olive seeds*, shows that the extract contains compounds such as protein, phenols, free sugars and fatty acids, oleic and linolenic acids. In view of these components it is clear that the majority of organic compounds containing oxygen atom and all have pairs of free electrons. The inhibitive property of *OS* may be explained by considering the adsorption of the *OS* extract molecule through the free electrons on the oxygen atom in these compounds and complex formation (surface chelation) on the corroding metal surface. These may be responsible for the formation of an oriented film layer, which essentially blocks discharge of H^+ and consequent dissolution of the metal ions and reduced the interaction between the aluminum with central corrosion and thus decrease the corrosion rate.

IV. CONCLUSION

The effect of acid concentration and the effect of addition the aqueous extract of *Olive seeds* (*OS*) on the corrosion of aluminum has been studied at 30 and 40°C. The following conclusions may be drawn:

1) The chemical (HE and ML) results showed that the corrosion rate of aluminum sample is increase with increasing acid concentrations (0.25-1.5) M and rising temperature (30-40)°C.

2) The aqueous extract of *OS* acts as good inhibitor for the corrosion of aluminum metal in 1 M HCl solution.

3) At 40°C, increasing of inhibition is found as concentration of the *OS* increased and *Inh. %* is higher than at 30°C.

4) The adsorption of *OS* extract molecules on aluminum metal surface follows Langmuir adsorption isotherm, the values of $K_{ads.}$, E_a indicate chemical adsorption.

5) It appears from SES and EIS methods that the presence of *OS* extract an adsorbed film on aluminum surface was contained due to adsorption of *Olive Seeds* components on aluminum surface which lead to corrosion inhibition. This results confirms with that obtained from mass loss and hydrogen evolution studies.

Table1: Corrosion rates of aluminum in different concentrations of HCl acid at 30°C

C (M)	0.01M	0.25M	0.5M	0.75M	1M	1.5M
R_{ML} x10^5 (ml.cm^{-2}min)	0.134	0.232	0.936	1.667	2.090	12.911
R_{HE} x10^2 (g.cm^{-2}min)	0	0.165	1.498	2.091	3.235	20.412

Table2: Corrosion rates of aluminum in 1M HCl at different temperatures.

Corr. Rate	30°C	40°C	50°C	60°C	70°C
R_{ML} x10^5	2.09	4.56	7.32	9.71	10.89
R_{HE} x10^2	3.235	7.806	11.15	15.00	15.68

Table3: Corrosion rates and inhibition efficiencies of aluminum in 1M HC l in the presence of different concentrations of an inhibitor (*OS* extract) at 30°C.

$C_{Inh.}$ %v/v	0.0	1	3	4	5	7	10
R_{ML} x10^5	2.090	1.104	1.109	0.320	0.289	0.278	0.051
R_{HE} x10^2	3.235	1.664	1.746	0. 387	0.253	0.248	0.037
$Inh._{ML}$%	--	47.18	46.94	84.69	86.17	86.70	97.56
$Inh._{HE}$%	--	48.56	46.03	88.04	92.18	92.33	98.86

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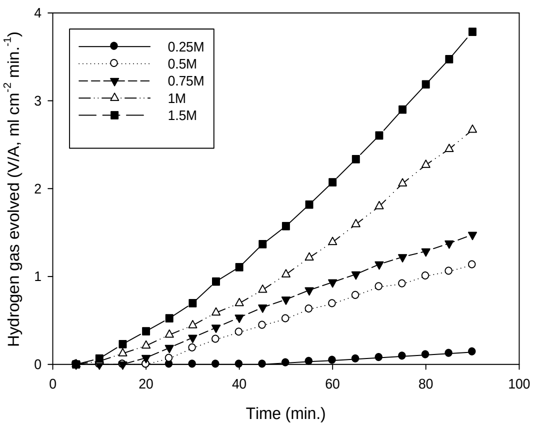

Fig. 1: Corrosion of aluminum in different concentrations of HCl at 30°C.

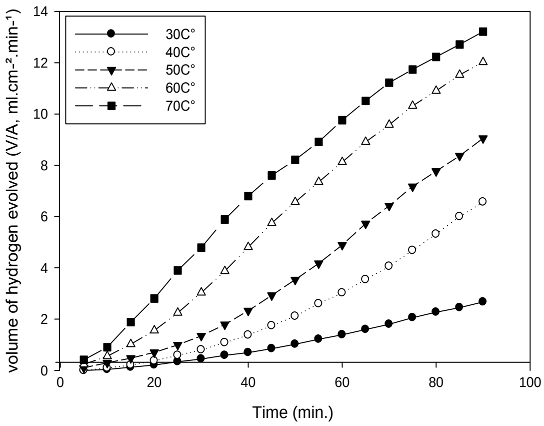

Fig. 2: Corrosion of aluminum in 1M HCl at different temperatures.

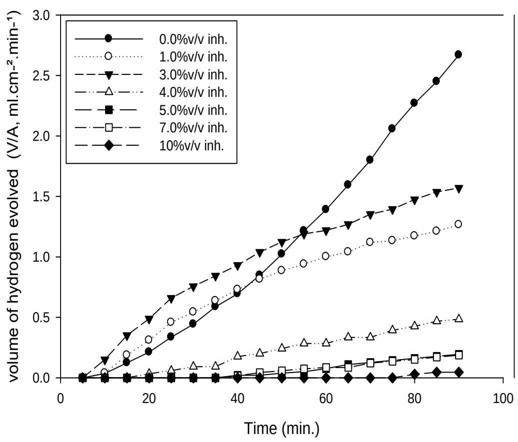

Fig. 3: Corrosion of aluminum in 1M HCl and different concentrations of inhibitor (*OS* extract) at 30°C.

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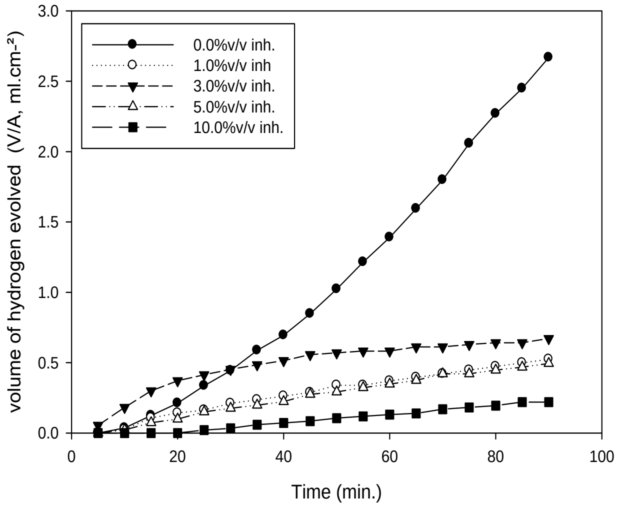

Fig. 4: Corrosion of aluminum in 1M HCl and different concentrations of inhibitor (*OS* extract) at 40°C.

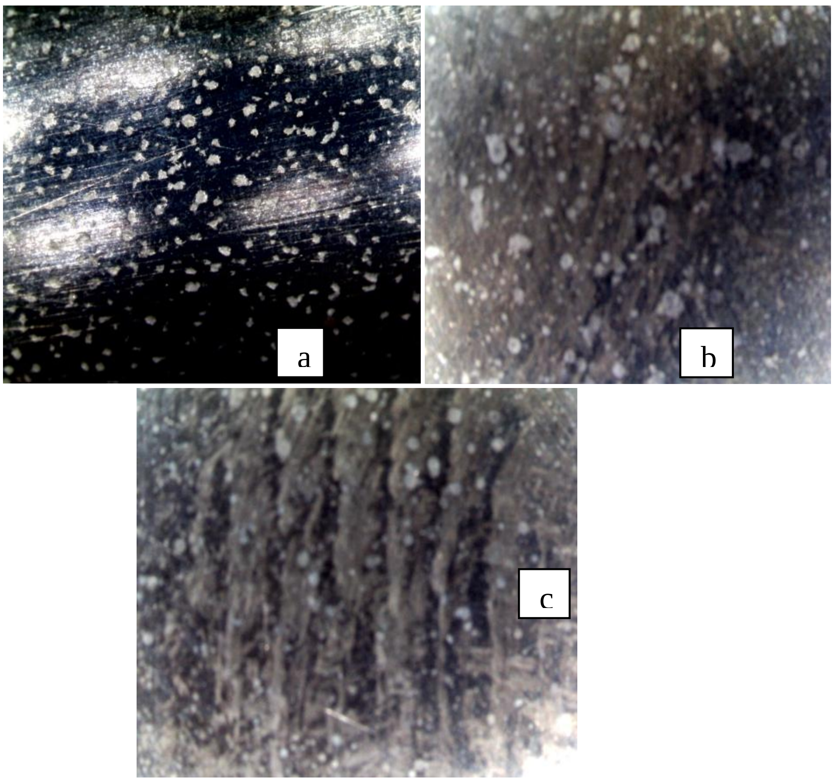

Fig. 5: SEM photographs of aluminum sample, (a) after corrosion, (b) in presence of 3% v/v *Aloe* extract (lower concentration) and (c) in presence of 10% v/v (higher concentration) of *OS* extract in 1 M HCl solution at 30° C.

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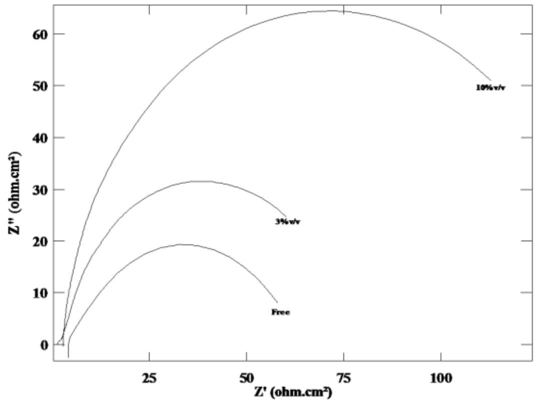

Fig. 6: Nyquist plots for aluminum corrosion in 1 M HCl in presence of 3% v/v (low concentration) and 10% v/v (high concentration) of *OS* extract at 30° C.

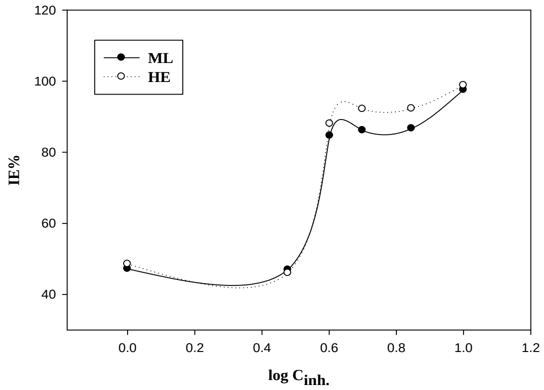

Fig. 7: The relation between IE.% and log $Ci_{nh.}$ for aluminum corrosion in 1 M HCl in the presence of different concentrations of aqueous extract of *OS* at 30° C.

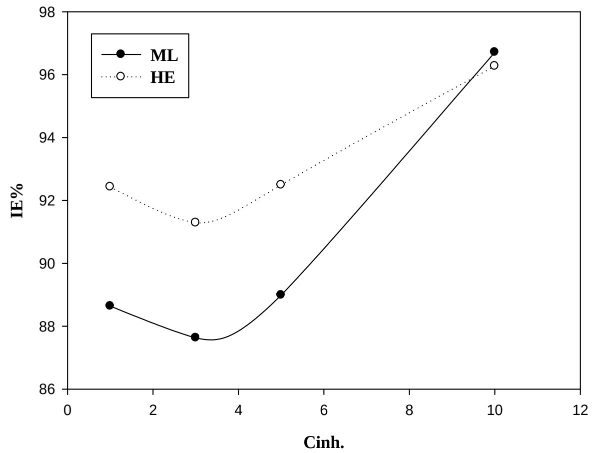

Fig.8: The relation between IE.% and log $C_{inh.}$ for aluminum corrosion in 1 M HCl in the presence of different concentrations of aqueous extract of *OS* at at 40^{0}C.

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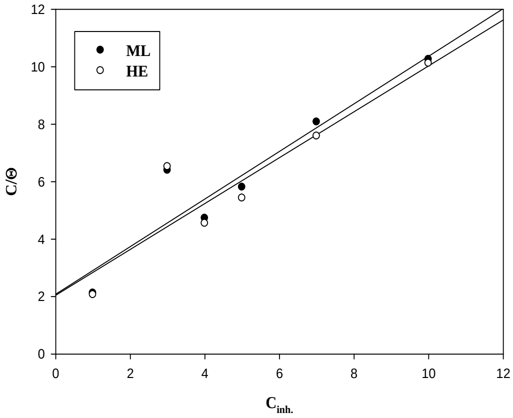

Fig. 9: Langmuir relationship for the adsorption of aqueous extract of *OS* on aluminum surface in 1 M HCl at 30^0 C

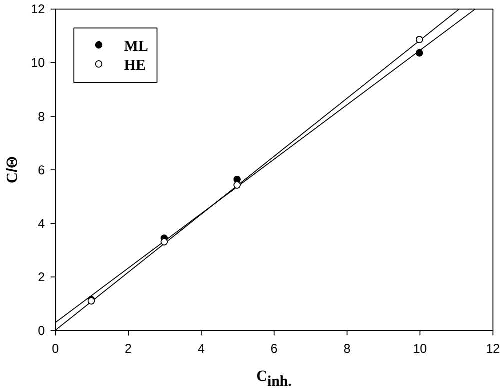

Fig.10: Langmuir relationship for the adsorption of aqueous extract of *OS* on aluminum surface in 1 M HCl at 40^0C .

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