J Biochem Tech (2018) 9(3): 10-16
ISSN: 0974-2328

Corrosion Prevention of Aluminum in Acidic Media Using Cydonia Vulgaris Leaves Extract as Environmentally Friendly Corrosion Inhibitor

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Received: 04 June 2018 / Received in revised form: 27 July 2018, Accepted: 02 August 2018, Published online: 15 August 2018

Abstract

Corrosion inhibition of aluminum in ($1\ M\ H_3PO_4$) was investigated in absence and presence of Cydonia Vulgaris leaves (CVL) extracts as friendly corrosion inhibitors. The effect of temperature and inhibitor concentration at immersion time of ($3h$) was studied using weight loss method. The obtained results revealed that Cydonia Vulgaris leaves(CVL) extracts inhibited aluminum in (H_3PO_4) and decreased the rate of corrosion. By increasing the concentration and temperature of the inhibitor, its efficiency increased. Higher inhibition efficiency for aluminum corrosion of (95.51%) was obtained at a higher level of inhibitor concentration and temperature. The adsorption of Cydonia Vulgaris leaves(CVL) extracts was found to obey Langmuir adsorption isotherm model. The values of the free energy of adsorption were more than ($-20\ kJ/mol$) which was indicative of a mixed mode of physical and chemical adsorption. Activation enthalpy (ΔH^*), and activation entropy (ΔS^*) of aluminum corrosion was found to be ($43.7254\ kJ\ mol^{-1}$) , ($-0.1804\ \mathrm{kJ\ mol}^{-1}K^{-1}$) and ($20.2732\ \mathrm{kJ\ mol}^{-1}$), ($-0.1921\ \mathrm{kJ\ mol}^{-1}K^{-1}$) in the absence and presence of the extract; respectively.

Keywords: Adsorption, Corrosion, Inhibitor Efficiency, Aluminum alloy, Phosphoric Acid, SEM, FTIR.

Introduction

Aluminum has been one of the most widely used metals, in competition with steel, and increasingly utilized in the fields of architecture, transportation, and public works (Al-Saeed et al., 2016). Aluminum and its alloys are low cost and remarkable materials in industrial technology because of their light weight, high thermal and electrical conductivity as well as high resistance to corrosion in a wide variety of corrosive environments (Hassan & Zaafarany, 2013). It forms a protective invisible passive oxide film on its surface upon exposure to an aqueous solution, but this oxide film does not offer sufficient protection against strong acidic, and alkaline solutions because of its solubility at these conditions, which increases the rate of corrosion (Gaballah et al., 2017).

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Aluminum and alloys of aluminum play a vital role in the automobile industries, aerospace applications, construction industries such as frames and facades; Aluminum is a good reflector for visible light as well as heat, and that along with its low weight makes it an ideal material for decorative and functional sees (Kumar, 2017; Ahmed, 2015).Phosphoric acid is a major industrial chemical, which is widely used for acid cleaning and electorpolishing of aluminum, even though, the dissolution rate of aluminum in phosphoric acid is low compared to the dissolution in hydrochloric or sulphuric acid, it does corrode aluminum and its alloys. Phosphoric acid is also used in pickling delicate, costly components and precision items where recruiting after pickling has to be avoided (Prabhu, 2013).Therefore, it is necessary to seek inhibitors for the corrosion of aluminum in (H_3PO_4).An extensive review of the literature has revealed that very little attention has been paid to inhibition studies on aluminum alloy in H_3PO_4 (Ameer et al., 2012).Corrosion inhibitor was popular for protecting alloys and steel structures in manufacturing. Such phenomenon could be found in various kinds of alloy surfaces bringing about the main economic damages in the manufacturing sector. Al-Okbi (2018) summarized the effect of inhibitors on the corrosion of aluminum and aluminum alloys in various media, and the degeneration in stainless steels and aluminum alloys materials because of their exposure to corroding environments. Inhibitors have been used to reduce the corrosion of these alloys in deferent corrosive media, and increase their durability. The use of corrosion inhibitors is necessary in cases like the exposure of Al alloys to acid or salt solution. Synthetic chemical inhibitors, commonly chromates, are toxic to life in the environment upon disposal or leaks and their handling may also be hazardous to health and safety (Al-alkawi et al., 2018; Al-Haj-Ali et al., 2014). The presence of heteroatoms in the structure of inhibitor molecules such as $O,\ N,\ S,\ P$, enhances the adsorption process and inhibition efficiency (Bataineh et al., 2013). Plenty of organic and inorganic compounds have been used by various researchers as corrosion inhibitors protect the dissolution of this oxide film (Chaubey et al., 2017). The mechanism of the organic inhibitor is usually an adsorption process. The heteroatom in the inhibitor's molecule acts as an active site for the adsorption to take place. The physical and chemical properties of inhibitor molecule influence the adsorption process, and they are related to the electronic density of donor atoms, and the possible steric effects. The aqueous extract of leaves contains various chemical constituents such as the leaves of A . vasica (AV) that contain alkaloids vasicine, N-oxides of vasicin, vasicinone, deoxyv asicine and maiontone (Khadom et al., 2018; Chaubey et al., 2015). In the present work, *Cydonia Vulgaris* leaves, which are very common, available and cheap plants in Diyala governorate/Iraq were extracted and tested to control the corrosion of aluminum in (H_3PO_4) solution at a different operating condition. This natural material has been used as a green and environmentally friendly material that can substitute the artificial ones.

Experimental

Preparation of aluminum coupons for anti-corrosion study

Aluminum samples were used in weight loss tests. The chemical compositions on weight bassis of these samples were as follows; Si (0.0481%), Fe (0.0684%), Cu (0.0035%), Mn (0.0017%), Mg (0.0006%), Cr (0.0002%), Ni (0.0011%), Zn (0.0054%), Ti (0.0012%), Ga (0.0078%), V (0.0062%) and the remainder is Al. The aluminum sheets with the thickness of ($1cm$) were cut into the samples of dimension ($3cm\ x3cm$) with a hole drilled on their sides for easy suspension in the corroding solution (Okek et al., 2015). The samples were polished by using emery paper with different grades ($220, 400, 600, 800, 1000, 1500$ and 2000). Then the samples were rinsed with flow of tap water followed by distilled water. They were then dried by electric dryer followed by immersing in methanol and acetone, dried again, and then stored in desiccators prior to use. The samples were weighted by ($4-digits$) electronic balance, and the dimensions were measured by an electronic vernier. The metal samples were completely immersed in($200\ ml$) corrosion solution of ($1\ M\ H_3PO_4$) contained in the beaker. They were exposed for a period of($3h$) at the desired temperature and inhibitor concentration. Then, the metal samples were cleaned, washed with running tap water followed by distilled water dried with clean tissue, then immersed in methanol and acetone and dried again. Weight losses were determined in presence and absence of inhibitor at($25, 35, 45$ and 55 ℃) and using ($1, 3, 5, 7, 9$ and$11\ ml/L$) inhibitor concentrations. Each test was repeated twice, and the average values were taken.

Preparation of plant extract for corrosion inhibition studies

Cydonia Vulgaris leaves (CVL) were collected from gardens, shade dried, ground and converted to powder. The extract was prepared by refluxing($10\ gm$) the powder in $100\ ml$ of ($1\ M\ H_3PO_4$) acid for ($3h$) and kept overnight, then filtered, and the filtrate was made up to ($50\ ml$) using the same acid, and this was taken as a stock solution, and portions with the volumes of (1,3,5,7,9 and 11 ml) /liter in (H_3PO_4) were used as a corrosion solution.

Preparation of Corrosion Solution

The solution of ($1\ M\ H_3PO_4$) was prepared by dilution of phosphoric acid of analytical grade (85%) using distilled water. The test solutions were freshly prepared before every experience, by directly adding the extract to the corrosion solution.

Scanning electron microscope (SEM)

The scanning electron microscope model ($TESCAN$, $Vega\ III$) made in Czech Republic was used to examine the morphology of aluminum surface in the absence and presence of *Cydonia Vulgaris* leaves (CVL) extracts, after ($3\ h$) immersion period in ($1\ M\ H_3PO_4$) at (55 ℃).

Fourier transform infrared spectroscopy ($FTIR$)

$FTIR$spectra were recorded using Perkin - Elmer 65 spectrophotometer made in Germany. The film was carefully removed and mixed thoroughly with KBr , and ($FTIR$) spectra were recorded. The spectrometer was used to evaluate the molecular structure, and active group of the inhibitor of (CVL).

Results and Discussion

Weight loss measurement

From the change in the weight of specimens, the corrosion rate was calculated using the following relationship (Al-Dokheily et al., 2014).

$$C_{Rcorr}\ (\text{mpy}) = \frac{534.W}{D.T.A} \quad (1)$$

And the obtained corrosion rate thus was used in the calculation of E_a, ΔS^*, ΔH^*, $where$; W, weight loss in milligram,D : density of specimen (g/cm^3), A: area of specimen in a square inch ($noting\ that\ 1\ in^2 = 6.5416\ cm^2$), and the exposure time was in hours,

$$mpy = mils\ per\ year\ (mill = 1inch/1000).$$

While inhibition efficiency was calculated using the equation below:

$$\%\text{IE} = \frac{\text{CR}_\circ - \text{CR}_\text{i}}{\text{CR}_\circ}\ \text{x}\ 100 \quad (2)$$

where CR_0 and CR_i are the corrosion rates in the absence and presence of various concentrations of inhibitor; respectively. Corrosion rate and inhibitor efficiency were evaluated at different operating conditions. The results have been presented in Table1. It was clear that corrosion rate increased when the temperature and the inhibitor concentration decreased.

Table 1- Corrosion rate (mpy) and inhibitor efficiency $(\%\ IE)$ and surface coverage (θ) as a function of temperature and inhibitor concentration.

Time (3 h)					
No.	$C_{inh}/(ml/L)$	Temperature / (℃)	$C_R/(mpy)$	θ (surface coverage)	$\%\ IE$
1	Blank	25	149.513	0	0
2		35	347.681	0	0
3		45	574.276	0	0
4		55	838.325	0	0
5	1	25	35.118	0.7652	76.52
6		35	65.065	0.8134	81.34
7		45	89.544	0.8444	84.44
8		55	93.928	0.8887	88.87
9	3	25	26.417	0.8260	82.60
10		35	51.758	0.8507	85.07
11		45	75.946	0.8690	86.90
12		55	82.410	0.9026	90.26
13	5	25	24.903	0.8347	83.47
14		35	47.662	0.8656	86.56
15		45	71.234	0.8758	87.58
16		55	74.011	0.9119	91.19
17	7	25	22.293	0.8521	85.21
18		35	41.787	0.8805	88.05
19		45	53.578	0.9074	90.74
20		55	66.361	0.9211	92.11
21	9	25	17.715	0.8869	88.69
22		35	29.980	0.9141	91.41
23		45	46.329	0.9209	92.09
24		55	52.750	0.9381	93.81
25	11	25	15.996	0.8956	89.56
26		35	29.327	0.9179	91.79
27		45	35.703	0.9390	93.90
28		55	37.854	0.9551	95.51

Effect of temperature and thermodynamic parameters calculations

The effect of temperature on the rate of corrosion of aluminum in $(1\ M\ H_3PO_4)$ containing a different concentration from investigated inhibitors was tested by the weight loss measurements over a temperature range from $(25-55℃)$. The effect of increasing temperature on the corrosion rate and $(\%\ IE)$ were obtained from the weight loss measurements. The results presented in Table 1 revealed that the rate of corrosion increased as the temperature increased, and decreased as the concentration of these compounds increased. The activation energy E_a of the corrosion process was calculated (Khadom et al., 2015) using equation 3 from the slope of the plot of $\ln K$ aginst $1/T$ which is $-E_a/T$.

$$K = A \exp\left(-\frac{E_a}{RT}\right) \tag{3}$$

Where K is the rate of corrosion, A is the Arrhenius constant, R is the gas constant, and T is the absolute temperature.

Figure. (1). presents the Arrhenius plots in the presence and absence of inhibitors. Similar curves were obtained in the presence of other inhibitors' concentrations, but not shown. E_a values determined from the slopes of these linear plots have been shown in Table 2. The linear regression (R^2) was close to 1 which indicated that the corrosion of aluminum in $1\ M\ H_3PO_4$ solution can be elucidated using the kinetic model. Table (2) also showed that the value of E_a inhibited solution was higher than that for the uninhibited solution, suggesting that the dissolution of aluminum was slow in the presence of inhibitor and can be interpreted as a chemical adsorption. It has been known from Equation (3) that the higher E_a values would lead to the lower corrosion rate. This was due to the formation of a film on the aluminum surface, serving as an energy barrier for the aluminum corrosion. Enthalpy and entropy of activation $\Delta H^*, \Delta S^*$ of the corrosion process were calculated from the transition state theory equation :

$$CR = \frac{R\ \ T}{N\ \ h} \exp\left(\frac{\Delta S_{act}}{R}\right) \exp\left(-\frac{\Delta H_{act}}{RT}\right) \tag{4}$$

Where (h) is Planck's constant, and (N) is Avogadro's number $6.022\ x10^{23}$. Equation (4) can be plotted as $\ln(CR/T)$ versus $(1/T)$ to calculate the activation enthalpy and entropy from slope $(-\frac{\Delta H_{act}}{R})$ and intercept $n\ (\frac{R}{N\ \ h}) + (\frac{\Delta S_{act}}{R})$; respectively. Figure (2) presents the transition plots in the presence and absence of the inhibitors. Similar curves were obtained in the presence of the other inhibitors, but not shown. The data have been given also in Table 2. The positive signs of ΔH^* reflected the endothermic nature of the aluminum dissolution process. Large and negative values of ΔS^* implied that the activated complex in the rate-determining step represented an association rather than a dissociation step, meaning that the decrease in disordering took place ongoing from reactants to the activated complex (Fouda et al., 2013).

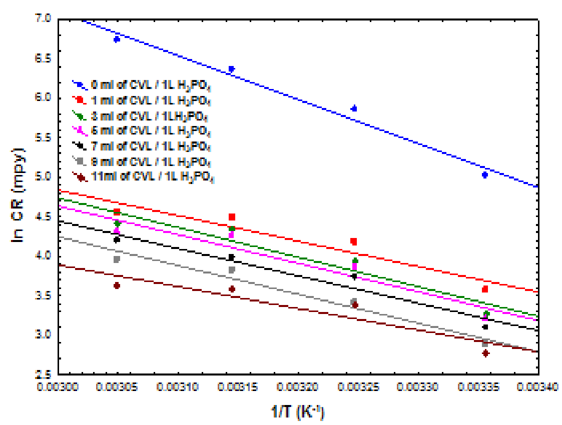


Fig. 1- Arrhenius plots for aluminum corrosion rates $(1\ M\ H_3PO_4)$ in absence and presence of different concentrations of CVL.

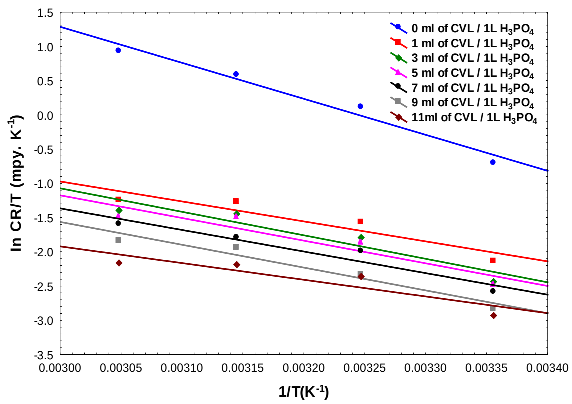


Fig. 2- Transition state plots for aluminum corrosion rates (1 M H_3PO_4) in absence and presence of different concentrations of CVL .

Table 2- Activation parameters .

C (ml/L)	E_a (kJ/mol)	$\Delta H^*(kJ/mol)$	$\Delta S^*(kJ/mol.K)$
0	46.323	43.7254	-0.1804
1	26.859	24.2614	-0.1897
3	31.140	28.5425	-0.1883
5	30.118	27.5207	-0.1887
7	28.812	26.2154	-0.1894
9	30.324	27.7270	-0.1890
11	22.870	20.2732	-0.1921

2 e Activation

Effect of inhibitor concentration

The inhibition efficiency of aluminum with different concentration of Cydonia Vulgaris leaves (CVL) extract in $1M$ H_3PO_4 at room temperature have been presented in Table 1. From the tables, it was clear that the corrosion rate decreased with an increase in inhibitor concentration. The corrosion inhibition was enhanced with the inhibitor concentration. This behaviour was due to the fact that the adsorption and coverage of the inhibitor on the aluminum surface was increased with the inhibitor concentration. The high inhibitive performance of Cydonia Vulgaris suggested a higher bonding ability of the inhibitor on aluminum surface (Preetha & Selvarani, 2017).

Adsorption isotherms

Organic compounds as green corrosion inhibitors decreased the corrosion of the metal through the adsorption on the metallic surface followed by forming a protective layer. In order to understand the mechanism of corrosion inhibition, the adsorption behaviour of the adsorbate on the aluminum surface must be known. The information on the interaction between the inhibitor molecules and the metal surface can be provided by adsorption isotherm. The degree of surface coverage ($\theta = IE/100$) for different concentrations of inhibitor was evaluated from weight loss measurements. Three models were suggested to study the kinetics of adsorption of (CVL) on the metal surface. Langmuir adsorption isotherm was obtained by Equation (5), Freundlich adsorption isotherm was measured by Equation. (6), and Temkin adsorption isotherm was assessed by Equation (7). It was found that the data best fitted the Langmuir adsorption isotherm.

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \tag{5}$$

$$\theta = K_{ads}\, C^n \tag{6}$$

$$\exp(-2a\theta) = K_{ads} C \tag{7}$$

where, θ is the degree of surface coverage,C is the concentration of the inhibitor, and K_{ads} is the adsorptive equilibrium constant and a is the molecular interaction parameters. Equations (5), (6) and (7) could be drawn as shown in Figure (3), (4), and (5). The Gibbes standard free energy of adsorption of the organic inhibitor was estimated by means of Equation. (8)

$$K_{ads} = \frac{1}{1000} \exp\left(\frac{-\Delta G^{\circ}{}_{ads}}{RT}\right) \tag{8}$$

Where $R = 8.314\, J.mol^{-1}$' is the universal gas constant, 1000 is the concentration of water in the corrodent solution (ml/L), while T is the absolute temperature in Kelvin. From Table (3), ($\Delta G^{\circ}{}_{ads}$), the values were negative in all the models used and lied in $(-19.561\ to\ -23.323) kJmol^{-1}$. It was clearly observed that the acid concentration increased the values of ($\Delta G^{\circ}{}_{ads}$) which revealed that the most efficient inhibitor showed more negative values, ($\Delta G^{\circ}{}_{ads}$)with a decrease in the inhibition efficiency and increase in acid concentration with a physical adsorption. This suggested that there was a strong adsorbtion on the metal surface. Generally, the values of ($\Delta G^{\circ}{}_{ads}$) more negative than $(-20\, kJmol^{-1})$ indicated the physical adsorption, while those more negative than $(-40\, kJmol^{-1})$ indicated the chemical adsorption. The values($\Delta G^{\circ}{}_{ads}$) obtained in this experiment was less negative than $(-20\, kJmol^{-1})$ also supported the physisorption process (Udom et al., 2017; BinYehmed et al., 2018; Desai, 2018).

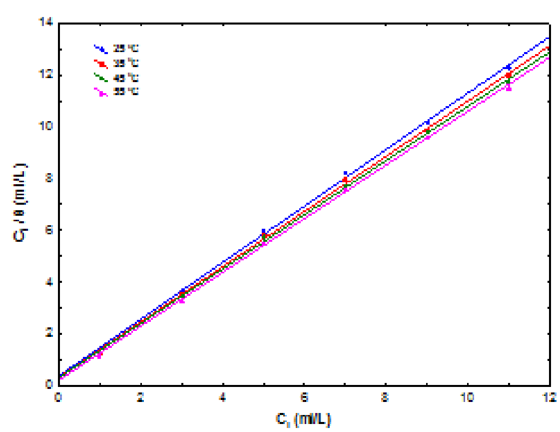


Fig. 3- Langmuir adsorption isotherm of aluminum in (1 M H_3PO_4) under different conditions.

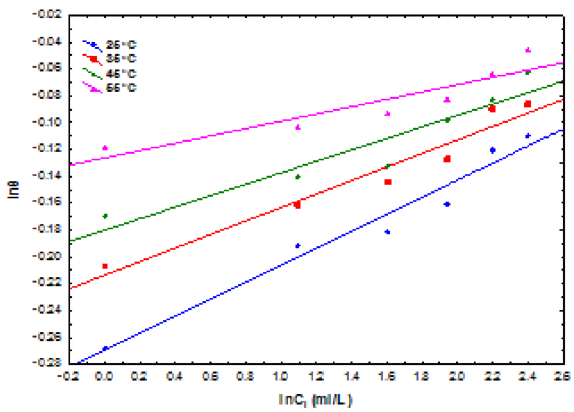


Fig. 4- Freundlich adsorption isotherm of aluminum in ($1\ M\ H_3PO_4$) under different conditions

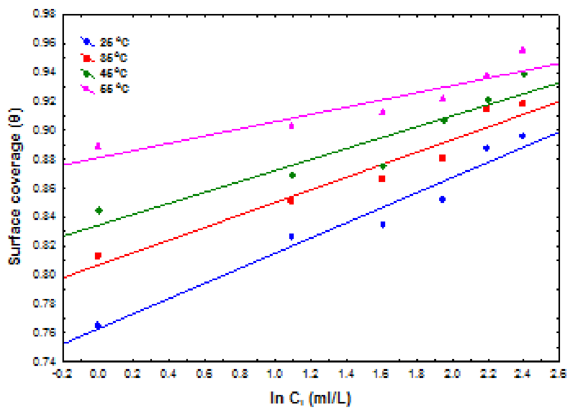


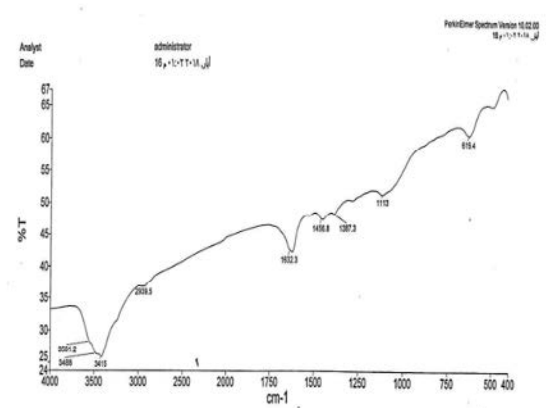
Fig. 5- Temkin adsorption isotherm of aluminum in ($1\ M\ H_3PO_4$) under different conditions.

Table 3- Adsorption parameters for corrosion of aluminum in inhibited ($1\ M\ H_3PO_4$).

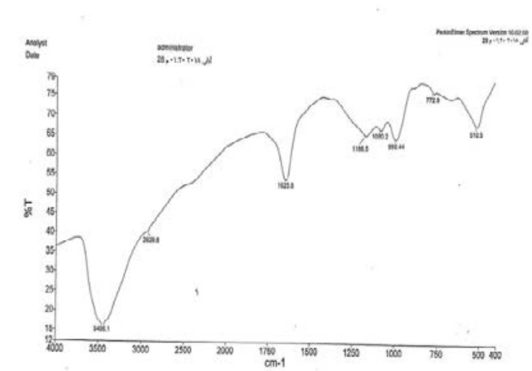
T, K	Langmuir adsorption isotherm			Freundlich adsorption isotherm			Temkin adsorption isotherm		
	K_{ads} (ml/L)	ΔG°_{ads} (ml/L)	R^2	K_{ads} (ml/L)	$n^{"}$	R^2	K_{ads} (ml/L)	a	R^2
298	7.9319	-19.561	0.9988	1.3123	0.063	0.9629	0.7627	0.052	0.956
308	8.1174	-20.786	0.9991	1.5441	0.050	0.9464	0.8067	0.043	0.9380
318	8.1771	-21.561	0.9990	1.7153	0.042	0.8925	0.8344	0.037	0.8843
328	8.5528	-23.323	0.9993	2.0698	0.027	0.8500	0.881	0.025	0.8422

FT-IR Spectroscopy Analysis

$FTIR$test was performed on the Cydonia Vulgaris leaves' (CVL) powder. The inhibitor was prepared by using (H_3PO_4) and the corrosion product (adsorbed film) was prepared by using $FTIR$ spectroscopy. Absorption frequencies were the values recorded from the ($FTIR$) spectra shown in Figure (6. a & b).The functional groups assigned from Cydonia Vulgaris leaves(CVL), the prepared inhibitor and the adsorbed film, were those functional groups found to participate in the adsorption and inhibition of various inhibitors which have been reported. The shift of these frequencies from the inhibitor to the adsorbed ones could be attributed to the interaction of inhibitor functional groups with the aluminum metal surface for the adsorption to take place. For example,($O-H$) vibration shifted from $3415\ cm^{-1}$ to $3406.1\ cm^{-1}$ in aluminum that of ($C-H$) from $2939.5\ cm^{-1}$ to $2929.6\ cm^{-1}$ in aluminum that of ($C=C$) from $1632.3\ cm^{-1}$ to $1625.8\ cm^{-1}$ in aluminum that of ($C-O$) from $1387.3\ cm^{-1}$ to $1166.5\ cm^{-1}$ in aluminum (Leelavathi & Rajalakshmi, 2013; Sangeetha et al., 2012).



(A)

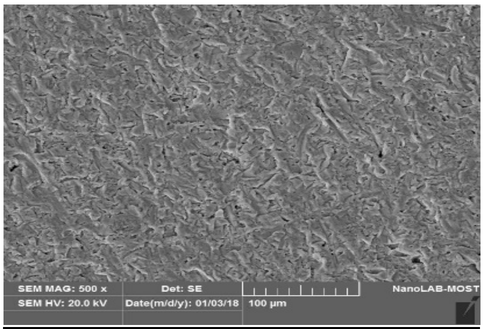


(B)

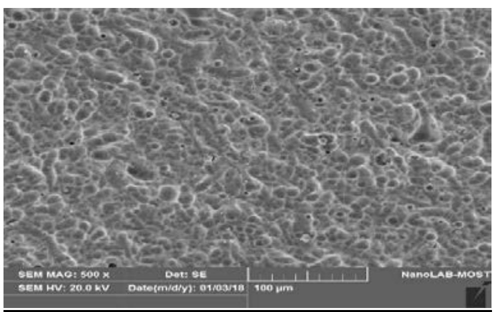
Fig. 6- $FTIR$ spectra of CVL ($11\ ml/L\ and\ 55℃$)
(A) CVL before contact with aluminum (B)CVL after contact with aluminum.

Surface Profile Analysis Using (SEM)

The scanning electron microscope (SEM)was used to evaluate the conditions of the aluminum surface in contact with $1\ M\ H_3PO_4$ solution in the absence and presence of (CVL) supporting the findings of the present work. The clean samples were immersed in the blank in $1M\ H_3PO_4$ in absence and presence of (CVL) for (3h); the images have been shown in Figure (7A). And, Figure(7B) represents the uncorroded surface of aluminum coupons without pits, but some scratches which occurred during filing. Uninhibited coupon with deep pits++ (some of them were overlaid by the lines in order to become easily seen) showed that the corrosion has taken place (see Figure. 7C). The inhibited coupon which difference with the profile of aluminum coupons was indicated above, proved the formation of the adsorbed layer film which acted as a barrier to the acid corrosiveness (Kasuga et al., 2018).



A



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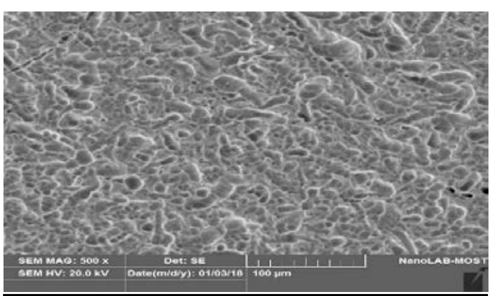

C

Fig. 7- SEM images of aluminum metal ($\boldsymbol{A}$) sample before immersion (B) sample after immersion in ($1\ M\ H_3PO_4$) solution ($\mathbf{55}$℃)($\boldsymbol{C}$) sample after immersion in $1\ M\ H_3PO_4$solution with inhibitor ($11\ ml/L\ at$ 55℃) .

Conclusion

The acidic extract of (CVL) acted as a good and efficient anticorrosion material for the corrosion of aluminum in the ($1\ M\ H_3PO_4$). The inhibition efficiency increased with the inhibitor concentration, and the maximum inhibition efficiency was (95.51 %)at the optimum concentration of (11 ml/L at 55°C). The adsorption of plant extract on the surface obeyed Langmuir adsorption isotherm. The negative sign of the free energy of adsorption indicated that the adsorption of the (CVL) on the metal surface was a spontaneous process and a combination of physical and chemical adsorption.$FTIR$ analysis showed that (CVL) plant extracts contained the mixtures of compounds, i.e. amides, amines, aromatic and organic acids. The investigations of the SEM figures showed that there was a severe damage on aluminum surface in the absence of the inhibitor. There were less pits and cracks observed in the inhibited surface. It confirmed that the metal surface was covered with the (CVL) molecules, and a protective inhibitor layer was formed.

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