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Phoenix Dactylifera L. Extracts as Green Corrosion Inhibitor for Aluminum in Acidic Medium

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***Abstract*: The present study investigates corrosion inhibition of aluminum under 1 N acidic condition using male and female *Phoenix Dactylifera L.* leaves extracts by weight loss measurement in the temperature range 313-333K. The efficiency of inhibitor increases with increase in inhibitor dosage from 8 to 40 g L^{-1} and decreases with increase in temperature from 30 to 60^{0}C. The inhibitor efficiency of male and female extracts are 88.70 % and 95.16% respectively at inhibitor dosage 40 g L^{-1}. Six, adsorption isotherm namely Langmuir, Freundlich, Temkin, Frumkin, Flory Huggins and El-awardy were tested, Langmuir isotherm best fitted to experimental data. The values of Gibbs free energy of adsorption G_{ads} for male and female extracts were found to be -15.76 KJ $mole^{-1}$ and -29.80 KJ $mole^{-1}$ respectively indicating physisorption of inhibitor molecules on aluminum surface.**

***Keywords*: *Phoenix Dactylifera L.*, Corrosion, Aluminum, Langmuir, Physisorption.**

I. INTRODUCTION

Aluminum possesses prevalent applications due to its high strength to weight ratio, eminent electrical and thermal conductivity and reasonably lower price (Klodian,2017). Corrosion is the process of deterioration of materials characteristics due to is chemical or electrochemical reaction with surrounding (Klodian, 2019). Corrosion inhibitors are added in minor quantity on metal surfaces or to the corrosive medium to decrease the liability of being affected by corrosion (Alan, 2020). Due to toxicity issues and complications associated with manufacturing process of synthetic corrosion inhibitors, use of plant extracts as corrosion inhibitors is focused nowadays. Addition of plant extracts to the corrosive media is beneficial over prevailing synthetic inhibitors due to their cost effectiveness, abundant availability, easy manufacturing processes and bio-degradable properties (Shi, 2016) . The restricted use of polymeric substances as corrosion inhibitors is due to their low mechanical strength (David, 2013). The limitation of sol-gel coatings on metals for corrosion prevention is due lack of availability of systematic protocol to evaluate stability and feasibility of sol-gel coatings (Duhua, 2009) . Different plant extracts such as *Dendrocalamus brandisii* (Xianghong, 2012), *Citrullus Colocynthis* (Rajkiran, 2011), *Ananas Sativum* (Ating, 2010), *Carica Papaya* (Chaubey, 2018), *Calotropis procera*, *Calotropis gigantea* (Sudesh kumar, 2013), *Prosopis Laevigata* (Ramirez, 2013) have been studied for corrosion inhibition of aluminum in acidic conditions. *Phoenix Dactylifera L.* commonly known as Date palm is cultivated in the area of 16000 km^2 of Kachchh region. The price of date fruits range from 10-400 INR per kilogram or more (Shah,2014). But date palm leaves finds no potential applications. *Phoenix Dactylifera L.* is dioeceous plant i.e. it possess separate male and female trees (Chincheng, 2017). In the present study, male and female date palm leaves individually were initially defatted with petroleum ether and then subjected to extraction with ethanol. The resultant ethanol extract was evaluated for its ability to inhibit corrosion of Al plates under acidic (1 N HCl) conditions by gravimetric batch experiments and analyzed for different classes of phytochemical present in it. Further, physisorption of inhibitor molecules on the surface of aluminum was confirmed by thermodynamic and adsorption isotherm parameters.

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II. MATERIAL AND METHODS

A. Preparation and Analysis of Phoenix Dactylifera L. extracts:

Male and female *Phoenix Dactylifera L.* leaves were collected in the month of December. They were washed with distilled water, dried under shade, powdered and further utilized for the experiment. 20 gm of male and female leaves powder was defatted with 200ml petroleum ether using Soxhlet apparatus followed by extraction with ethanol in same proportion. The resultant extract was utilized for corrosion inhibition experiment, preliminary phytochemical screening and GC-MS analysis.

B. Preparation of corrosion media and specimen:

A 36% w w^{-1} HCl with density 1.18 gm cm^{-3} was successively diluted with distilled water to get 1M HCl. Al plates utilized in the study were obtained from local market. Further, they were resized to 4cm*4 cm and further used in the experiment.

C. Gravimetric batch experiment and effect of temperature:

In gravimetric batch experiment, specimen Al plates was immersed in corrosion media 1M HCl containing 0,2,4,6,8,10 ml of male and female ethanol extracts respectively at 303K (room temperature). Weight loss was recorded in each case after time interval of 1 hour. To determine the effect of temperature, the above mentioned experiment was repeated at temperatures 313, 323 and 333K respectively. Corrosion rate (CR), inhibitor efficiency (IE) and surface coverage (θ) in case was calculated using following equation 1-3 [13]:

$$\textbf{Corrosion Rate (CR)} = \Delta\mathbf{W}/\mathbf{A}t \quad (1)$$

$$\text{Inhibitor efficiency (IE)} = \frac{W'' - W'}{W''} \quad (2)$$

$$\text{Surface coverage } \theta = 1 - \frac{W'}{W''} \quad (3)$$

Where ΔW = weight loss after time interval t hours, W'' = corrosion rate in the absence of inhibitor, W'= corrosion rate in the presence of inhibitor, A= area of cross section of the plate 16 cm^2. The units of corrosion rate and inhibitor efficiency are gm $cm^{-2}.h^{-1}$ and % respectively.

D. Surface Analysis:

The surface morphology of specimen Al plates before and after corrosion was determined SEM.

E. Adsorption Isotherms:

The effectiveness of corrosion inhibitor mainly confide on its capability to adsorb on the metal surface [14]. Adsorption isotherms provide information concerning the nature of interaction between the inhibitor and surface of Al substrates [15, 16]. In this study, Langmuir, Freundlich, Temkin, Frumkin, Flory-Huggins and El-awardy adsorption isotherms are investigated. All these adsorption isotherm can be expressed in linear mathematical form as:

Langmuir adsorption isotherm model (Chakravarthy,2014 ; S. Arul, 2018):

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \quad (4)$$

Freundlich adsorption isotherm model (Olatunde,2020):

$$\log\theta = \log K_{ads} + n\log C \quad (5)$$

Temkin adsorption isotherm model (Joseph,2012):

$$\theta = -\frac{2.303\log K_{ads}}{2a} - \frac{2.303\log C}{2a} \quad (6)$$

Frumkin adsorption isotherm model (Rehim,2016):

$$\log\frac{C\theta}{1-\theta} = 2.303\log K_{ads} + 2\alpha\theta \quad (7)$$

Flory- Huggins adsorption isotherm model (Beniken,2018):

$$\log\frac{\theta}{C} = \log K_{ads} + x\log 1-\theta \quad (8)$$

El- awardy adsorption isotherm model (Gobara, 2017):

$$\log\frac{\theta}{1-\theta} = \log K + y\log C \quad (9)$$

Here, $K_{ads} = K^{\frac{1}{y}}$

III. RESULTS AND DISCUSSION

A. Preliminary Phytochemical Screening of Phoenix Dactylifera L. extracts:

Table 1: Different classes of phytochemicals present in *Phoenix Dactylifera L.* extracts:

Phytochemical Name	Male extract	Female extract
Proteins & Amino acids	++	+
Carbohydrates	+++	++
Phenols	++	+
Flavanoids	+++	++
Phlobatannins	-	+
Tannins	++	++
Lactones	+	+++
Quinones	-	+

Steroids	++	-
Terpenoids	++	-
Cardiac glycosides	+	++
Resins	-	++
Anthocyanins	-	-
Oils and fats	++	++
Saponins	+	++

(- = negative or absent, += weakly positive, ++= moderately positive, +++= strongly positive)

The above result shows that male and female extracts are enriched with flavanoids, Saponins, tannins, glycosides, oils and fats which are adsorbed on the metal surface through lone pair of electrons on heteroatom O, N and S leading to the formation of shielding metal inhibitor complex. This causes diminution in corrosion rate because of reduced metal acid contact (Beniken,2018 ; Ikeuba,2015).

B. *Identification Of Chemical Constituents of Male and Female Phoenix Dactylifera L. Leaves by GC-MS:*

Table 2: GC-MS analysis of male *Phoenix Dactylifera L.* extract:

Sr.no	**Retention time (Min)**	**Relative %**	**Compound Name**
1	12.73	14.78	n-tetradecane
2	15.21	17.57	n-tridecane
3	18.68	20.21	Dibutyl phthalate
4	18.96	16.70	n-Hexadecanoic Acid
5	22.77	16.92	Dioctyl adipate

Table 3: GC-MS analysis of female *Phoenix Dactylifera L.* extract:

Sr. no	**Retention time (Min)**	**Relative %**	**Compound Name**
1	12.43	6.89	Diphenyl ether
2	12.73	16.03	n-tetradecane
3	15.214	11.76	n-heptadecane
4	18.68	38.22	Dibutyl phthalate
5	18.95	18.80	n-Hexadecanoic Acid

Dibutyl phthalate is major constituent of both male and female extracts with relative proportions 20.21 % and 38.22% respectively. Dibutyl phthalate finds application as insect repellant, plasticizer, solvent and fixative for perfume, and textile lubricating agent (David,2005). Also, male and female extracts consists of 16.70% and 18.80% of n-hexadecanoic acid,commonly known as palmitic acid respectively. Palmitic acid possesses potential anti-bacterial and anti-fungal properties (Agoramoorthy, 2007).

Palmitic acid possesses potential anti-bacterial and anti-fungal properties (Onuegbu,2013).

C. *Effect of inhibitor concentration on corrosion rate and inhibitor efficiency:*

Table 4: Corrosion rate and inhibitor efficiency of male *Phoenix Dactylifera L.* extracts 303K

Inhibitor dose C ($g\ L^{-1}$)	**Corrosion rate Gm $cm^{-2}\ h^{-1}$**	**Inhibitor efficiency (%)**
Blank	0.03875	----
8	0.01375	64.51
16	0.006875	82.25
24	0.00625	83.87
32	0.005	87.09
40	0.004375	88.70

Table 5: Corrosion rate and inhibitor efficiency of female *Phoenix Dactylifera L.* extracts 303K

Inhibitor dose C ($g\ L^{-1}$)	**Corrosion rate Gm $cm^{-2}\ h^{-1}$**	**Inhibitor efficiency (%)**
Blank	0.03875	----
8	0.01375	64.51
16	0.00875	77.41
24	0.00375	90.32
32	0.0025	93.54
40	0.001875	95.16

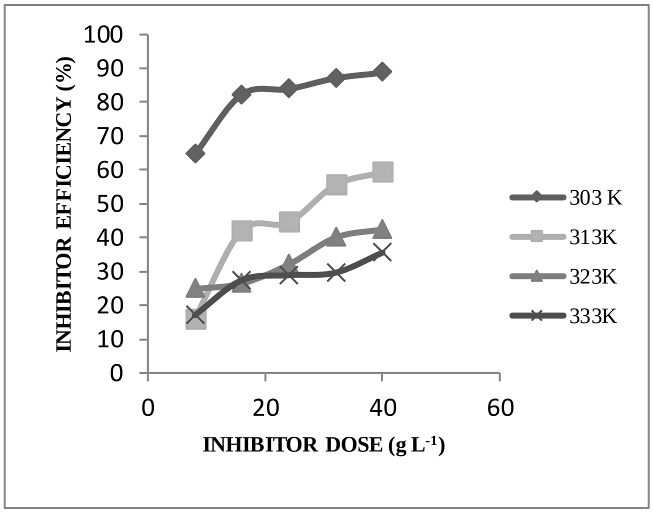

Figure 1: Variation in inhibitor efficiency of male extract with inhibitor dose at different temperature

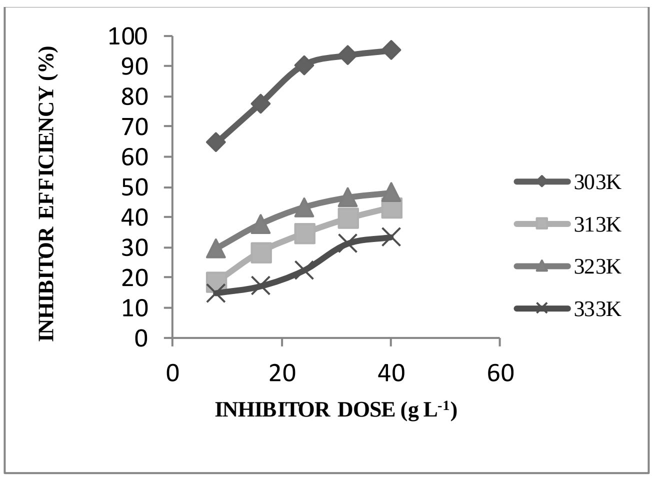

Figure 2: Variation in inhibitor efficiency of female extract with inhibitor dose at different temperature

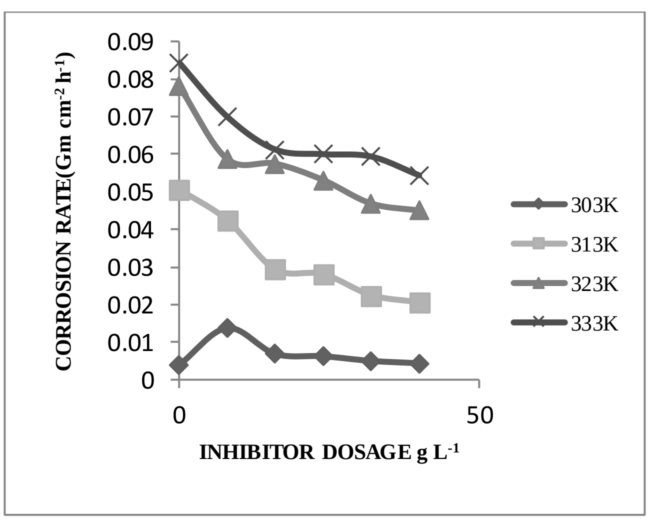

Figure 3: Variation in corrosion rate of male extract with inhibitor dose at different temperature

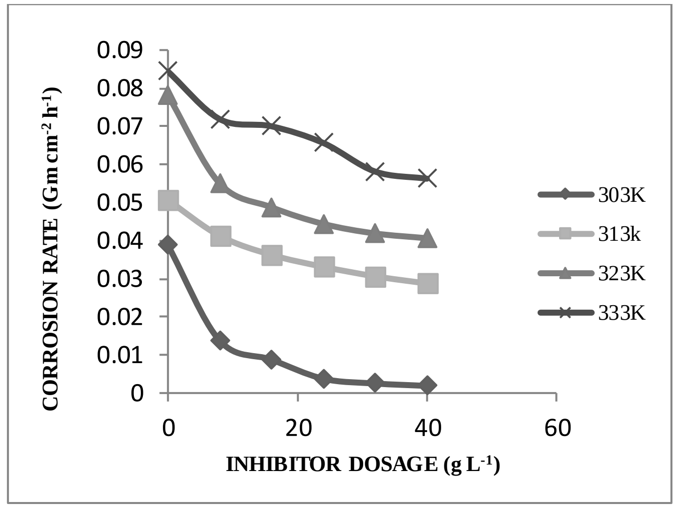

Figure 4: Variation in corrosion rate of female extract with inhibitor dose at different temperature

The inhibitor efficiency increases from 64.51 to 88.70% and 64.51 to 95.16% for male and female extracts respectively on increasing inhibitor concentration from 8 g L^{-1} to 40 g L^{-1}. At the same time corrosion rate declines from 0.03875 gm/cm^2 h to 0.004375 gm cm^{-2} h^{-1} and 0.001875 gm cm^{-2} h^{-1} for male and female extracts respectively. The increase in inhibitor concentration leads to increased adsorption of inhibitor molecules on the surface of Al due to which inhibitor efficiency increases and corrosion rate decreases (Krishnaveni,2014).

D. Adsorption isotherms:

Table 6: Adsorption isotherm parameters for female *Phoenix Dactylifera L.* extract at 303K

Adsorption isotherm	**R^2**	**Slope**	**Intercept**
Langmuir	0.997	0.91	5.335
Freundlich	0.969	0.255	-0.415
Temkin	0.973	0.465	0.227
Frumkin	0.976	5.731	-2.359
Flory-Huggins	0.955	0.551	-0.889
El-awardy	0.97	0.625	0.791

Table 7: Adsorption isotherm parameters for male *Phoenix Dactylifera L.* extract at 303K

Adsorption isotherm	**R^2**	**Slope**	**Intercept**
Langmuir	0.998	1.033	3.657
Freundlich	0.886	0.191	-0.345
Temkin	0.904	2.701	-0.877
Frumkin	0.955	5.241	-2.268
Flory-Huggins	0.927	1.119	-0.554
El-awardy	0.951	1.084	0.588

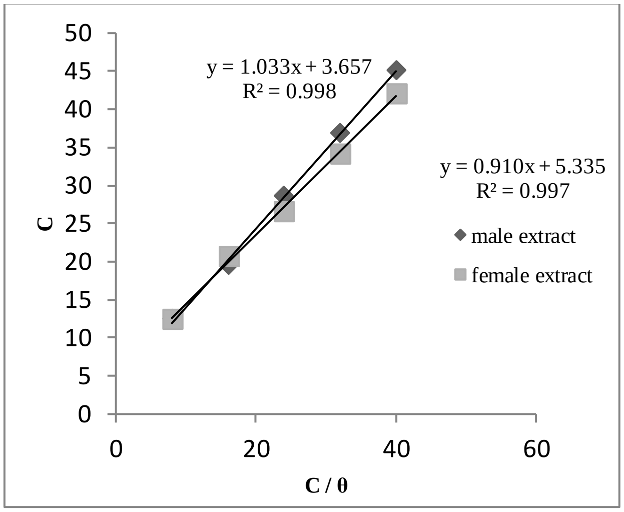

Figure 5: Langmuir adsorption isotherm plot for male and female *Phoenix Dactylifera L.* extracts

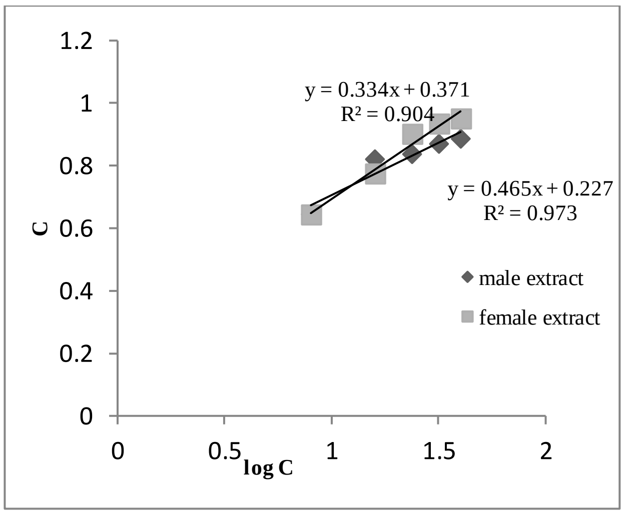

Figure 6: Freundlich adsorption isotherm plot for male and female *Phoenix Dactylifera L.* extracts

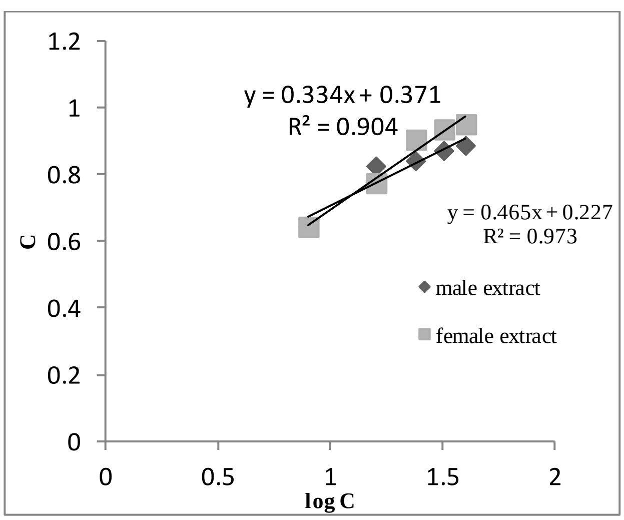

Figure 7: Temkin adsorption isotherm plot for male and female *Phoenix Dactylifera L.* extracts

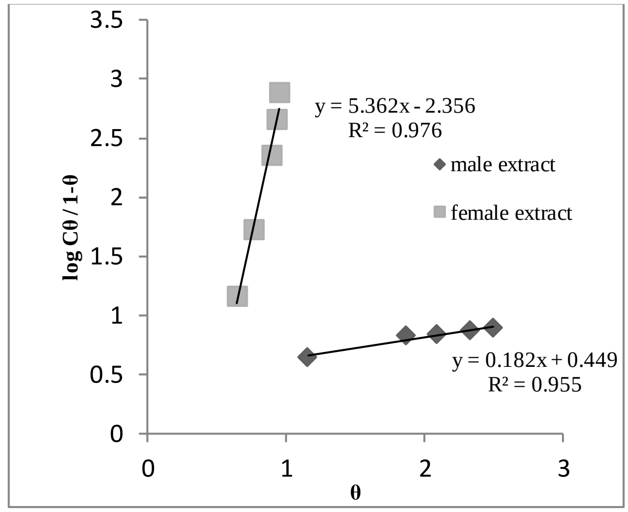

Figure 8: Frumkin adsorption isotherm plot for male and female *Phoenix Dactylifera L.* extracts

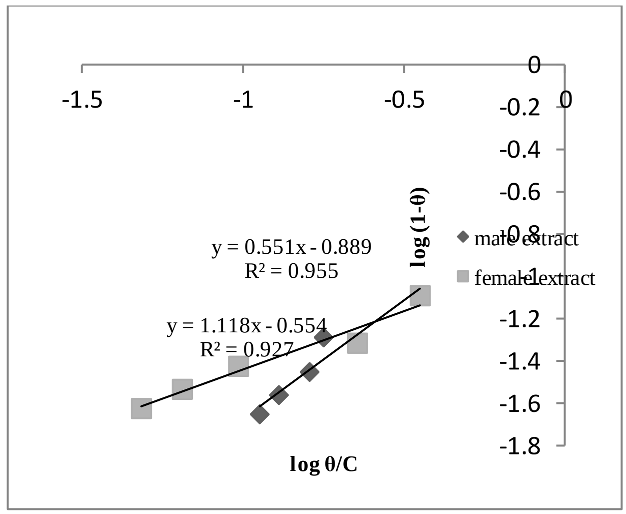

Figure 9: Flory Huggins adsorption isotherm plot for male and female *Phoenix Dactylifera L.* extracts

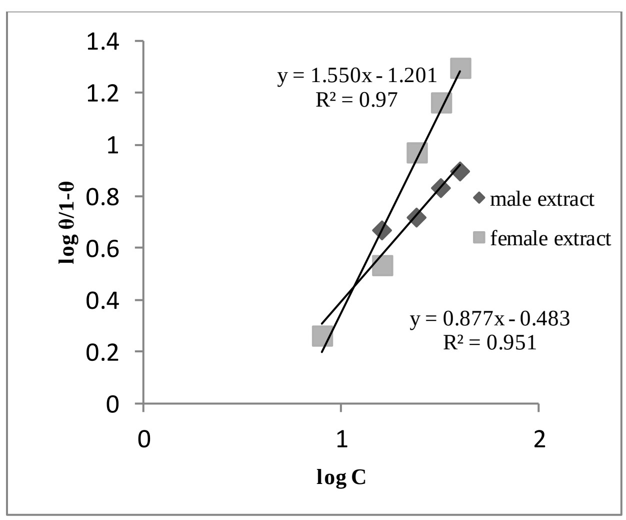

Figure 10: El-awardy's adsorption isotherm plot for male and female *Phoenix Dactylifera L.* extracts

From the table 4 and 5, it is evident that Langmuir adsorption model best describes the corrosion inhibition process of Al by male (R^2= 0.998) and female (R^2=0.997) *Phoenix Dactylifera L.* leaves. Langmuir adsorption isotherm only considers the interaction between the metal surface and inhibitor molecules neglecting the interactions in between the adsorbent molecules. The value of K_{ads} is related to the intercept of Langmuir plot as follows:

$$K_{ads} = \frac{1}{intercept} \quad (10)$$

The Gibbs free energy of adsorption was calculated using the equation [14]:

$$G_{ads} = -2.303\ RT \log(55.5\ K_{ads}) \quad (11)$$

Where G_{ads}= Gibbs free energy of adsorption, R= universal gas constant, T=absolute temperature, K_{ads} = adsorption equilibrium constant.

Table 8: Values of adsorption equilibrium constant and Gibbs free energy of adsorption for male and female *Phoenix Dactylifera L.* extract at 303K

Extract	K_{ads}	G_{ads}(KJ mole^{-1})
Male	0.273	-15.769
Female	0.187	-29.808

The negative values of G_{ads} for male and female extracts indicate spontaneous adsorption of inhibitor molecules on the surface of Al plates. The values of G_{ads} upto -20 KJ mole^{-1} are related with physisorption while -40 KJ mole^{-1} or lower than those are associated with chemisorption (Rehim, 2016) . The values of G_{ads} for male and female extracts at 303K are found to be -15.769 and -29.808 KJ mole^{-1} respectively indicating physisorption of inhibitor molecules on the surface of Al.

E. Effect of temperature:

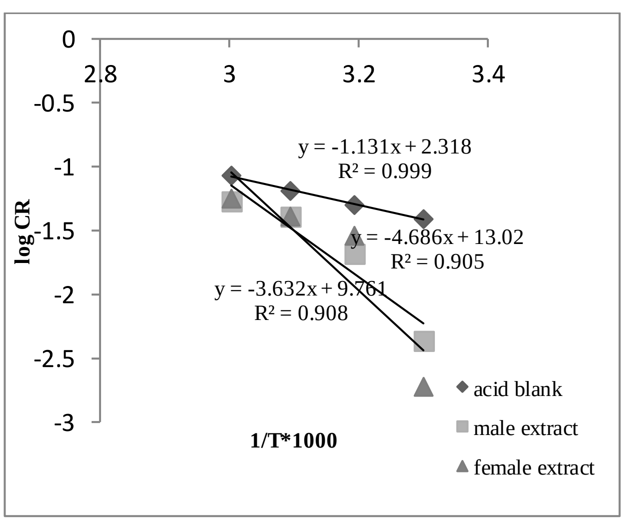

Figure 11: Arrhenius plot for blank (1 M HCl), male and female *Phoenix Dactylifera L.* extracts at dosage 40 g/L

From figure 3 and 4, it is evident that corrosion rate increases while inhibitor efficiency decreases with increase in temperature from 303K to 333K. Due to increase in temperature, the layer of inhibitor molecules assembled on the metal surface becomes less stable and hence inhibitor efficiency decreases. Arrhenius equation is used to evaluate activation energy of inhibitor and can be stated as follows (Bashir,2020):

$$\log CR = -\frac{E_a}{2.303RT} + \log A \quad (12)$$

Where CR= corrosion rate, R= universal gas constant, T=absolute temperature, E_a = activation energy, A = Arrhenius constant.

Table 9: Values of activation energy E_a (KJ mole^{-1}) calculated from Arrhenius plot

Sample	R^2	Activation energy E_a (KJ mole^{-1})
Blank	0.999	21.65
Male	0.908	69.54
Female	0.905	89.72

The activation energy for male and female extracts was found to 69.54 and 89.72 KJ mole^{-1} respectively which are higher than that of blank 21.65 KJ mole^{-1}. The higher values of activation energy in the presence of extracts signifies improved inhibitive action of extracts by increasing energy barrier required for the corrosion process (Ambrish Singh, 2016). The values of activation energy E_a greater than 80 KJ mole^{-1} are associated with chemisorption while lower than 80 KJ mole^{-1} are associated with physisorption (Eddy,2008). In the present study, for both male and female extracts, values of activation energy are lower than 80 KJ mole^{-1} indicating physisorption of inhibitor molecules on the metal surface. Also, higher activation energy of female extract as compared to male extract indicates better corrosion inhibition property of female extract as compared to male extract. The same has been confirmed from corrosion rate values for male and female extracts 0.003475 and 0.001875 gm cm^{-2} h^{-1} respectively.

In acidic solution, existence of positively charged Al surface is attributed to inflation of $Al\text{-}OH_2^+$ species in the media. The preliminary phytochemical screening of male and female extracts reveals the presence of flavanoids, Saponins, tannins, glycosides, oils and fats containing oxygen or nitrogen atoms in their molecules and they are regarded as centers of adsorption. Adsorption of these phytochemicals on the surface of aluminum results in barrier for mass and charge transfer. Hence , metal is protected from dissolution and corrosion in acidic media (Xianghong,2012; Ating,2012; Chaubey;2018).

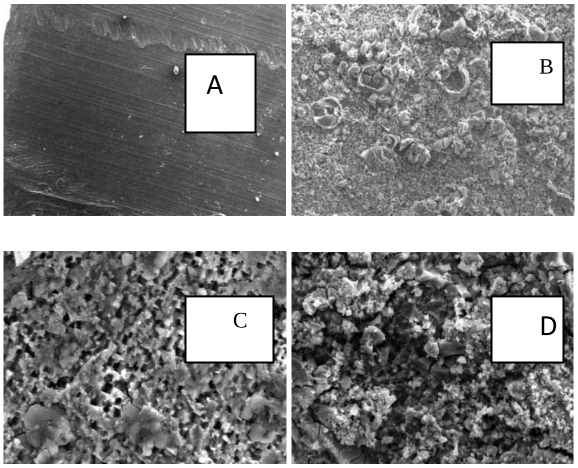

Figure 12: SEM images of (A) Al plate (B) corroded Al plate with 1M HCl (C) male extract as inhibitor (D) female extract as inhibitor

CONCLUSION

The present study concludes that both male and female *Phoenix Dactylifera L*. extracts are potent green corrosion inhibitors for Al in 1 N HCl. The corrosion inhibition process for both extracts well fitted to Langmuir adsorption isotherm indicating monolayer formation of inhibitor molecules on the surface of Al. Both male and female extracts are enriched with phytochemicals flavanoids, Saponins, tannins, glycosides, oils and fats which increase the activation energy barrier required for corrosion and hence lower corrosion rate(Onuegbu,2013). Physisorption of inhibitor molecules on the metal surface has been confirmed from the values of Gibbs free energy of adsorption and activation energy.

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