

# Corrosion Inhibition of Steel in Petroleum Medium by *Ficus Carica* Leaves Extract

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**ABSTRACT**— *This work involves study the corrosion inhibition of steel in petroleum medium using alcoholic extracted of Ficus carica leaves with four concentrations of extracted (1, 3, 5 and 7mL/L) at four temperatures over the range 50 – 80 °C. Inhibition efficiency IE% results show that 3 and 5 mL/L the best concentrations which they gave efficiencies in the range of 71 – 80%. Thermodynamic functions of adsorption processes were calculated from experimental polarization data and the interpretation of the results reveals that Ficus carica extract Ficus carica extract obey Langmuir adsorption isotherm. Polarization curves indicate that Ficus carica extract is a mixed type inhibitor. The small values of the equilibrium constant of the adsorption-desorption process  $K_{ads}$  refer to physical adsorption process. The values of free energy of adsorption  $\Delta G^o_{ads}$ , enthalpy  $\Delta H^o_{ads}$  and entropy  $\Delta S^o_{ads}$  were negative indicating the spontaneous and exothermic adsorption process as well as Ficus carica extract retained the metal ions and reducing the dissolution of steel and the activated complex in the rate determining step denotes an association rather than a dissociation step.*

**Keywords**— Ficus carica extract; inhibition of steel; Petroleum medium.

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## 1. INTRODUCTION

In refineries and petrochemical plants, the amount of water is usually small, but the corrosivity is high and is localized at regions where the aqueous phase contacts the metal. The water may contain dissolved hydrogen sulfide (H<sub>2</sub>S), carbon dioxide (CO<sub>2</sub>), and chloride ions (Cl<sup>-</sup>). Corrosion may occur even when the produced water content is as low as 0.1%, or corrosion activity may not begin until after several years of production. Refineries and petrochemical industries employ a variety of film-forming inhibitors to control wet corrosion. Most of the inhibitors are long-chain nitrogenous organic materials, including amines and amides. Water-soluble and water-soluble-oil-dispersible type inhibitors are continuously injected, or oil-soluble and oil-soluble-water-dispersible type inhibitors (batch inhibitors) are intermittently applied to control corrosion.

Film-forming inhibitors anchor to the metal through their polar group. The nonpolar tail protrudes out vertically. The physical adsorption of hydrocarbons (oils) on these nonpolar tails increases film thickness and the effectiveness of the hydrophobic barrier for corrosion inhibition. Because inhibitors are interfacial in nature, they are active at liquid-liquid and/or liquid-gas interfaces and can lead to emulsification. As a result, foaming is sometimes experienced in the presence of inhibitors [1].

A number of organic and synthetic compounds showed a good anticorrosive activity, most of them are highly toxic to both human and environment. These toxic affection have led to the use of natural products as anticorrosion agents which are eco-friendly and harmless. In 1930, plant extracts dried stems, leaves, seeds and other plants were used in H<sub>2</sub>SO<sub>4</sub> acid pickling baths. Animal proteins found in products of meat and milk industries were also used for retarding acid corrosion. Recently, many authors investigated the inhibition by green inhibitors using electrochemical method and FTIR spectra as well as adsorption isotherm calculations. Ambrish et al. studied the inhibition by the fruits extract of Shahjan (*Moringaoleifera*), Pipali (*Piper longum*) and Orange (*Citrus aurantium*) using weight loss, electrochemical impedance spectroscopy, potentiodynamic polarization and linear polarization techniques [2]. Extracts of kola plant and

tobacco in different concentrations were used as 'green' inhibitors [3]. Abu-Dalo et al. studied the inhibition effect of exudates gum from Acacia trees (Gum Acacia, GA) on the corrosion of mild steel in acidic media [4]. Shivakumar et al. tested the leaf extracts of centella asiatica (CE) as green corrosion inhibitor for mild steel in 0.5 M H<sub>2</sub>SO<sub>4</sub> [5]. Sangeetha et al. used an aqueous extract of asafetida as a corrosion inhibitor in controlling corrosion of carbon steel. The main constituent of this extract is umbelliferone [6]. Benali et al. studied the corrosion and inhibition behaviors of mild steel in sulfuric acid + 5% EtOH in the presence of tannin extract of Chamaerops humilis plant (LF-Ch) and potassium iodide (KI) [7]. Some quinazoline derivatives were studied on the corrosion of carbon steel in 2M HCl [8]. Rajam et al. evaluated the inhibition efficiency (IE) of an aqueous extract of garlic in controlling corrosion of carbon steel in well water in the absence and presence of Zn<sup>2+</sup> [9]. Iloamaeka et al. studied the use of leaves extract of Emilia sonchifolia and Vitex doniana as corrosion inhibitors of mild steel in 2.5M HCl medium [10]. Khaled and Ebenso investigated the inhibition performance of cerium sulphate tetrahydrate on the corrosion of steel in 1.0 M HCl solutions [11].

The aim of present work is attempt to inhibit the corrosion of steel in petroleum medium by ethanolic extracted of Ficus carica leaves in temperatures range 50 – 80°C. Potential–time measurements, Tafel plots were used to evaluate the efficiency of *Ficus carica* extract.

## 2. EXPERIMENTAL PART

### 2.1 Materials and solutions

Carbon steel was used in this work (chemical composition wt%: 0.121 C, 0.22 Si, 0.44 Mn, 0.014 P, 0.016 S, 0.041 Cr, 0.002 Mo, 0.022 Ni, 0.02 Al, 0.002 Co, 0.055 Cu and Fe remain) obtained by SpectroMAX. Cubic carbon steel (10x10x3mm) with a square surface area (1cm<sup>2</sup>) was used in all experiments. The specimen was mounted by hot mounting using formaldehyde (Bakelite) at 138°C for 8 minutes to insulate all but one side and made a hole on one side of electrical connection and then the mounted specimens has been grinded with SiC emery papers in sequence of 220, 400, 600, 800, and 1200 grit to get flat and scratch-free surface and polished to mirror finish using polish cloth and alpha alumina 0.5µm and 1µm, and then washed with distilled water, degreased with acetone and rinsed with distilled water. The base electrolyte was petroleum medium obtained from Iraqi oil refinery. Analysis of medium is listed in Table (1) obtained by many techniques. Extraction of *Ficus carica* leaves were achieved by washing the leaves by distilled water and then drayed and grounded. Ethanolic extract carried out by dissolving 5 gm of grounded leaves in 200 mL ethanol and then refluxed at 45°C. The obtained extract was filtered by using Whatmann filter paper and concentrated to 100 mL. Four volumes of ethanolic extract were used as inhibitor includes 1, 3, 5 and 7 mL/L at four different temperatures 50, 60, 70 and 80°C.

### 2.2 Electrochemical Measurements

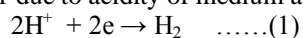
Electrochemical cell was composed of platinum counter electrode, prepared carbon steel specimen as working electrode and saturated calomel electrode (SCE) as a reference electrode according to ASTM standard cell G5-94 was used. The electrochemical behavior of carbon steel in inhibiting and uninhibited solution was studied by WINKING M Lab potentiostat by recording anodic and cathodic galvanodynamic polarization curves. Measurements were carried out by changing the electrode current automatically from -15 to +15 mA at scan rate 1 mA.sec<sup>-1</sup>, while open circuit potential recorded after immersion in test electrolyte for 600 sec. The linear Tafel segments of anodic and cathodic curves were extrapolated to the corrosion potential to obtain corrosion parameters.

## 3. RESULTS AND DISCUSSION

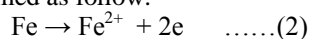
### 3.1 Corrosion Behavior

Potential – time measurements were achieved to evaluate the potential of the sample as a function of time in order to study the evolution of the film chemistry as it came to equilibrium with the solution for 600 sec. Figure (1) show the variation of potentials with time in petroleum medium in absence and presence four concentrations of *Ficus carica* extract. This figure indicates more regular changes in potential with time in presence of *Ficus carica* extract compared with absence of extract in petroleum medium in addition to get open circuit potentials E<sub>oc</sub> more noble than that observed in absence of inhibitor, i.e., the *Ficus carica* extract covered the metallic surface formed barrier at material/medium interface. The data of E<sub>oc</sub> are listed in Table (2) at different temperatures.

Figure (2) shows the Tafel plots of steel in petroleum medium in absence and presence of four concentrations of *Ficus carica* extract at four temperatures in the range 50 – 80°C. These curves show the cathodic and anodic regions, at cathodic sites, reduction of hydrogen can occur due to acidity of medium as follow:



While at anodic sites, ferrous ions may be formed as follow:

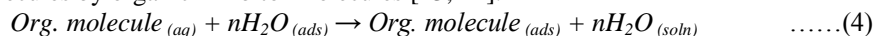


The data of polarization curves are listed in Table (2) which indicates that the corrosion potentials E<sub>corr</sub> shift toward either noble or active direction in the presence of inhibitor, i.e., *Ficus carica* extract acts as mixed inhibitor. The

values of corrosion current densities  $i_{corr}$  became lower than its values in absence of Ficus carica extract. The inhibition efficiency IE (%) can be calculated using the equation given below [12]:

$$IE\% = \frac{(i_{corr})_a - (i_{corr})_p}{(i_{corr})_a} \times 100 \dots\dots(3)$$

Where  $(i_{corr})_a$  and  $(i_{corr})_p$  are the corrosion current density ( $\mu A.cm^{-2}$ ) in the absence and the presence of the inhibitor, respectively. The best efficiencies were in presence of 3 and 5 mL/L of Ficus carica extract which ranged from 71 to 80%. On adsorption, most of the metal surface is covered by the adsorbed water molecules. The inhibitors react by replacing water molecules by organic inhibitor molecules [13, 14].



### 3.2 Adsorption Isotherm

Adsorption depends mainly on the charge and the nature of the metal surface, electronic characteristics of the metal surface, adsorption of solvent and other ionic species, temperature of corrosion reaction and on the electrochemical potential at solution-interface. The most frequently used isotherms include: Langmuir, Frumkin, Temkin, Flory-Huggins, Dhar-Flory- Huggins, Bockris-Swinkels and the recently formulated thermodynamic/kinetic model of El-Awady et al. [15, 16]. The establishment of adsorption isotherms that describe the adsorption of a corrosion inhibitor can provide important clues to the nature of the metal- inhibitor interaction. Adsorption of the organic molecules occurs as the interaction energy between molecule and metal surface is higher than that between the H<sub>2</sub>O molecule and the metal surface [17].

In order to obtain the adsorption isotherm, the degree of surface coverage ( $\theta$ ) for various concentrations of the inhibitor has been calculated according to equation:

$$\theta = \left[ 1 - \frac{i_{inh}}{i_{uninh}} \right] \dots\dots(5)$$

The plots of  $C/\theta$  against  $C$  for the inhibitor were straight lines (Fig. 3) indicating that Ficus carica extract obeys Langmuir adsorption isotherm given by the equation:

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \dots\dots\dots(6)$$

Where,  $K_{ads}$  is the equilibrium constant of the adsorption-desorption process,  $\theta$  is the degree of surface coverage and  $C$  is the molar concentration of inhibitor in the bulk solution. The linear regression coefficient close to unity, hence, adsorption of inhibitor followed Langmuir adsorption isotherm and  $R^2$  values were in the range  $0.979 \geq R^2 \geq 0.917$  as listed in Table (3). The Langmuir isotherm is based on the assumption that each site of metal surface holds one adsorbed species. Therefore, one adsorbed H<sub>2</sub>O molecule is replaced by one molecule of the inhibitor adsorbate on the steel surface. The apparent free energy of adsorption ( $\Delta G_{ads}^0$ ) is calculated from the relation [18]:

$$\Delta G_{ads}^0 = -2.303RT \log 55.5 K, \text{ where } K = \frac{\theta}{C(1-\theta)} \dots\dots(7)$$

The values of  $K_{ads}$  and  $\Delta G_{ads}^0$  are shown in Table (4). The enthalpy of adsorption ( $\Delta H_{ads}^0$ ) can be calculated from the Gibbs-Helmholtz equation:

$$\left[ \frac{\partial(\Delta G_{ads}^0/T)}{\partial T} \right]_p = -\frac{\Delta H_{ads}^0}{T^2} \dots\dots(8)$$

This equation can be arranged to the following equation:

$$\frac{\Delta G_{ads}^0}{T} = \frac{\Delta H_{ads}^0}{T} + K_{ads} \dots\dots(9)$$

The entropy of adsorption  $\Delta S_{ads}$  can be calculated by using following equation:

$$\Delta G_{ads} = \Delta H_{ads} - T\Delta S_{ads} \dots\dots(10)$$

The negative values of  $\Delta G_{ads}^0$  indicated the spontaneous adsorption of the Ficus carica extract and revealed strong interaction between inhibitor molecules and metal surface [16, 17]. The values of  $K_{ads}$  are relatively small indicating that the interaction between the adsorbed extract molecules and steel surface is physically adsorbed. This is also supported by lower negative ( $\Delta G_{ads}^0$ ) values for extract. Generally, values of ( $\Delta G_{ads}^0$ ) up to  $-20$  kJ/mol are consistent with electrostatic interactions between the charged molecules and the charged metal surface, which indicates physical adsorption [19]. The negative sign of  $\Delta H_{ads}^0$  in petroleum medium indicates that the adsorption of inhibitor molecule is exothermic process. Generally, an exothermic adsorption process signifies either physic- or chemisorption while endothermic process is attributable unequivocally to chemisorption [20], i.e., this result was good agreement with the results of  $K_{ads}$  and  $\Delta G_{ads}^0$ .

The value of  $\Delta S_{ads}^0$  is negative indicating that Ficus carica extract retained the metal ions and reducing the dissolution of steel in petroleum medium in presence of corrosion inhibitor as shown in Table (4). Also the negative values of  $\Delta S_{ads}^0$  implies that the activated complex in the rate determining step denotes an association rather than a dissociation step, meaning that a reduction in disordering takes place on going from reactants to the activated complex.

Generally, the inhibitive characteristics of such compounds derive from the adsorption ability of their molecules, with the polar group acting as the reaction center for the adsorption process. The resulting adsorbed film acts

as a barrier that separates the metal from the corrodent, and efficiency of inhibition depends on the mechanical, structural, and chemical characteristics of the adsorption layers formed under particular conditions.

### 3.3 Activation Energy

To elucidate the mechanism of inhibitor adsorption, it is necessary to establish the adsorption modes of the inhibiting species, whether, molecular or ionic. The predominant adsorption mode will be dependent on factors such as the extract composition, type of anion as well as chemical changes to the extract. The physical adsorption mechanism, obtained from electrostatic attractive forces between inhibiting organic ions or dipoles and the electrically charged surface of the metal due to the electric field existing at the metal/solution interface. A negative surface charge will favor the adsorption of cations whereas; anion adsorption is favored by positive surface charge. The apparent activation energies ( $E_a^*$ ) for the corrosion process in absence and presence of Ficus carica extract were evaluated from Arrhenius equation [21]:

$$\log C_R = \log A - \left[ \frac{E_a^*}{2.303 RT} \right] \dots (11)$$

where  $C_R$  is the corrosion rate ( $\text{g/cm}^2 \cdot \text{min}$ ),  $A$  is the constant frequency factor and  $E_a^*$  is the apparent activation energy,  $R$  is the gas constant ( $8.314 \text{ J} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$ ) and  $T$  is the absolute temperature. By plotting of logarithm of the corrosion rate of steel in petroleum medium in absence and presence of Ficus carica extract versus the reciprocal of absolute temperature range (323 – 353 K), give straight lines with slope equal to  $(-E_a^*/2.303R)$  is represented in Fig. (4). The data of activation energy are listed in Table (4-11) which demonstrate that, the presence of plant extracts in petroleum medium increase the values of  $E_a^*$  comparing to its uninhibited, these attributed to an appreciable decrease in the adsorption process of the inhibitors on the metal surface with increasing of temperature and a corresponding increase in the reaction rate because of the greater area of the metal that is exposed to the petroleum medium and corrosion inhibition occurred through physical adsorption [22].

## 4. CONCLUSION

Alcoholic extract of Ficus carica leaves was used as corrosion inhibitor for carbon steel in petroleum medium at four temperatures in the range 323 – 353 K. This study was performed using electrochemical method and indicates that Ficus carica extract was shifted corrosion potentials toward active or noble direction, e.i. this extract behave as mixed inhibitor, and decreased the corrosion current densities. This extract was physical adsorbed through the small values of equilibrium constant of adsorption and the negative values of free energy. The adsorption process was exothermic and characterized by decreasing in entropy due to activated complexes in the rate determining step denotes an association rather than a dissociation step.

## 5. REFERENCES

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**Table 1:** Analysis of petroleum medium.

<i>Analysis</i>	<i>Amount</i>
Total carbon	48.67%
Total hydrogen	8.73%
Total nitrogen	0.13%
Total sulfur	0.71%
pH	5.25
Elect. conductivity	300 $\mu$ S/cm
TDS	296 mg/L

**Table 2:** Corrosion parameters for polarization of carbon steel in petroleum medium in the absence and presence of alcoholic Ficus carica extract at four temperatures.

<i>Conc. of extracted</i>	<i>Temp. K</i>	<i>E<sub>oc</sub> mV</i>	<i>E<sub>corr</sub> mV</i>	<i>i<sub>corr</sub> <math>\mu</math>A.cm<sup>-2</sup></i>	<i>-b<sub>c</sub> mV.dec<sup>-1</sup></i>	<i>+b<sub>a</sub> mV.dec<sup>-1</sup></i>	<i>IE%</i>
<b>0</b>	323	-585	-759.8	497.68	1095.3	1087.7	-
	333	-570	-322.0	682.72	1632.2	1647.9	-
	343	-558	-496.2	694.09	2321.5	1957.9	-
	353	-375	-665.1	719.78	1606.6	1597.8	-
<b>1 mL/L</b>	323	-203	-390.6	217.40	449.7	432.0	56.31731
	333	-343	-572.0	221.63	466.6	656.6	67.53720
	343	-263	-493.2	266.68	472.2	360.1	61.57847
	353	-411	-467.6	295.78	521.6	505.7	58.90689
<b>3 mL/L</b>	323	-397	-733.0	129.27	652.8	588.4	<b>74.02548</b>
	333	-331	-600.3	145.31	516.1	448.2	<b>78.71602</b>
	343	-340	-479.3	166.63	535.1	442.3	<b>75.99303</b>
	353	-427	-639.3	198.32	457.2	547.3	<b>72.44714</b>
<b>5 mL/L</b>	323	-335	-148.2	142.34	99.6	85.8	<b>71.39929</b>
	333	+19	-7.1	153.12	587.8	177.0	<b>77.57206</b>
	343	-31	-293.8	169.68	347.3	379.3	<b>75.5536</b>
	353	-191	-517.9	143.70	344.1	453.4	<b>80.03557</b>
<b>7 mL/L</b>	323	-400	-546.8	257.44	904.2	953.3	48.27198
	333	-76	-533.9	265.60	438.0	596.0	61.09679
	343	-81	-601.2	252.49	389.5	621.8	63.62287
	353	-83	-531.9	224.99	428.0	601.9	68.74184

**Table 3:** The linear regression coefficients for adsorption of Ficus carica extract on steel surface in petroleum medium at different temperature.

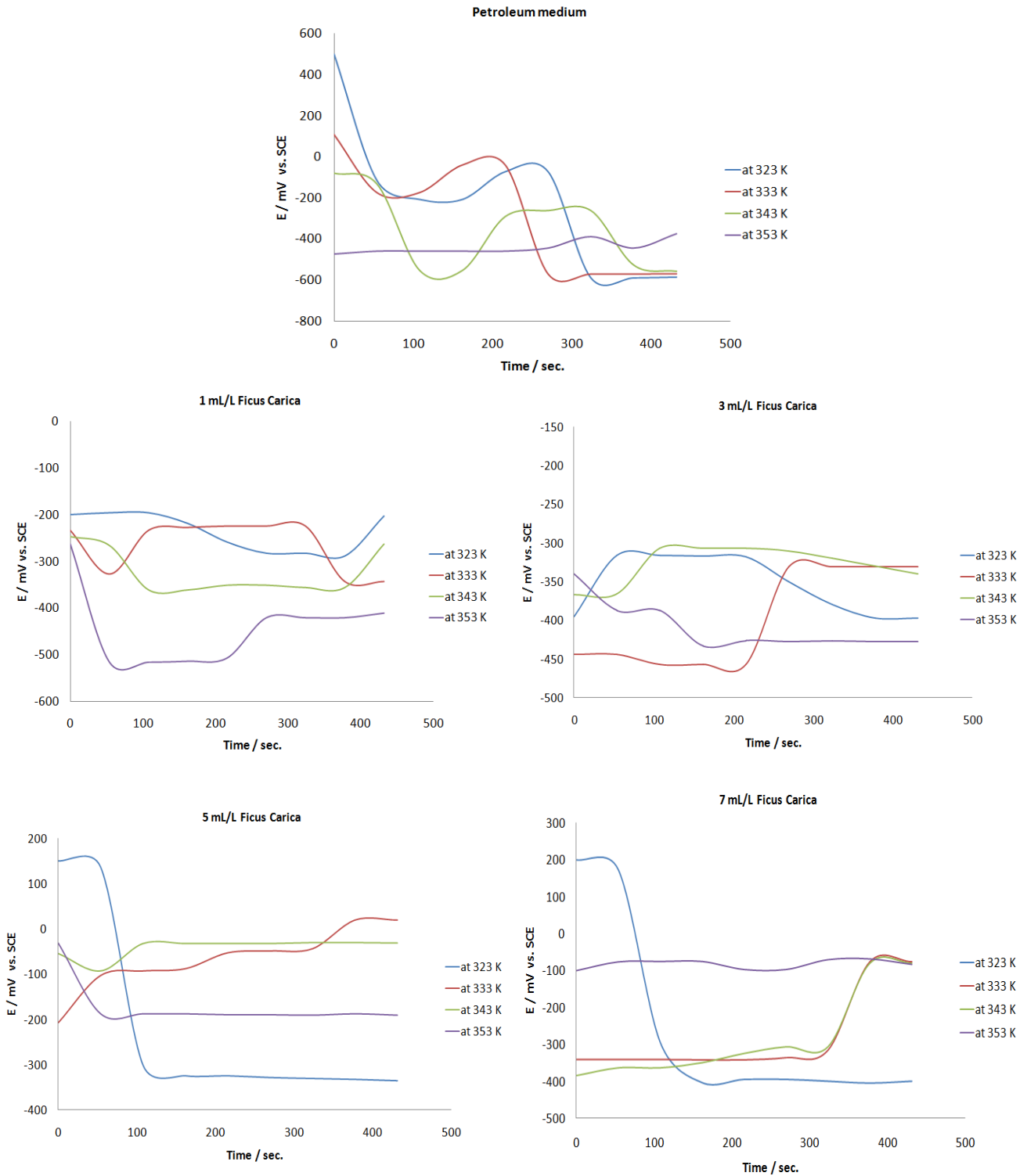
<i>Temp. /K</i>	<i>R<sup>2</sup></i>
323	0.917
333	0.963
343	0.976
353	0.979

**Table 4:** Thermodynamic functions for adsorption of Ficus carica extract in petroleum medium at four temperatures.

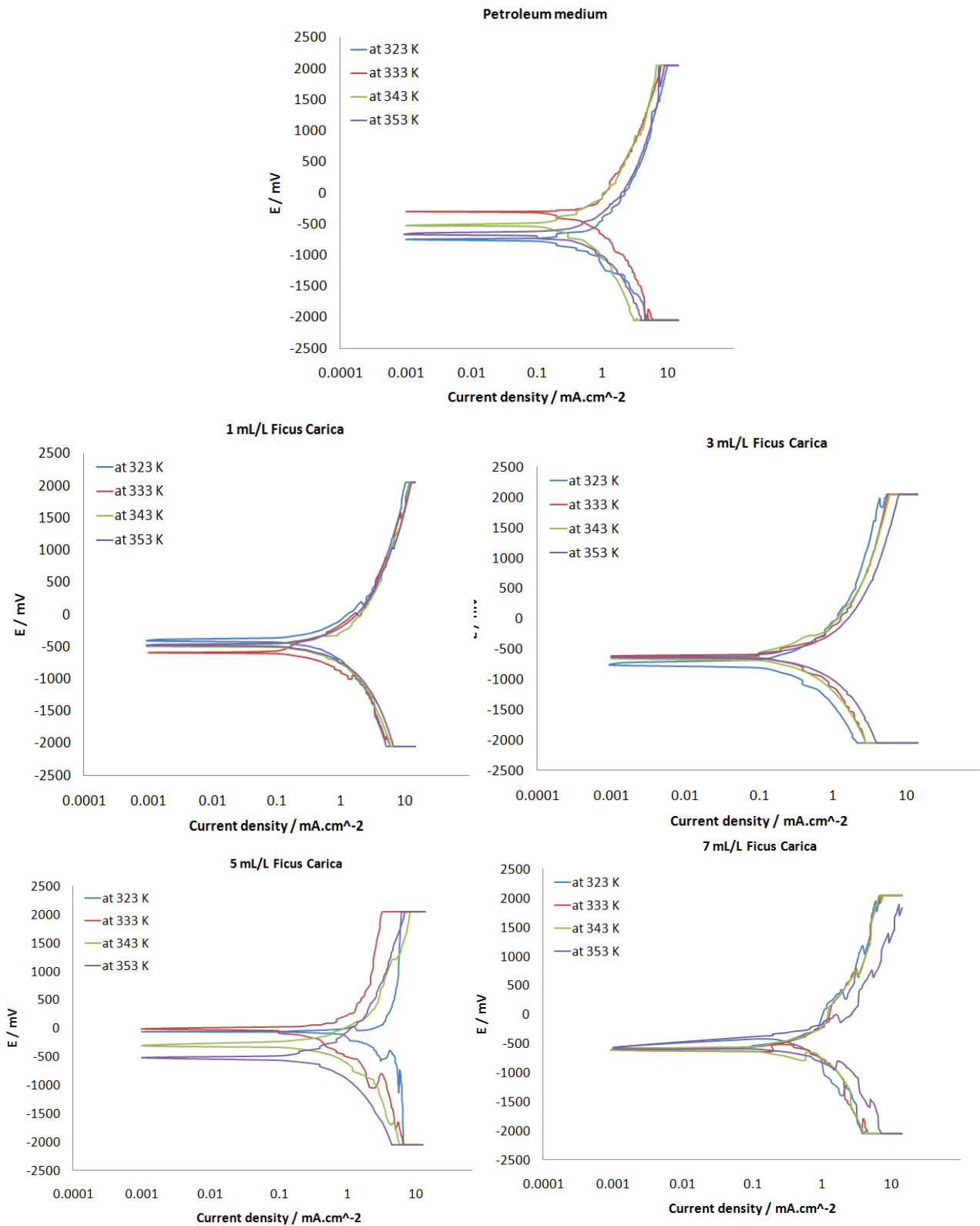
<i>Conc. of extracted</i>	<i>Temp. K</i>	<i>K<sub>ads</sub></i>	<i>ΔG<sup>o</sup><sub>ads</sub> kJ.mol<sup>-1</sup></i>	<i>ΔH<sup>o</sup><sub>ads</sub> kJ.mol<sup>-1</sup></i>	<i>ΔS<sup>o</sup><sub>ads</sub> kJ.mol<sup>-1</sup>K<sup>-1</sup></i>
1 mL/L	323	1.28922	-11.2161	-427.634	-1.28922
	333	2.08043	-12.8591	-705.642	-2.08043
	343	1.602676	-12.5176	-562.235	-1.60268
	353	1.433504	-12.5624	-518.589	-1.4335
3 mL/L	323	0.949952	-10.414	-317.248	-0.94995
	333	1.232788	-11.4422	-421.961	-1.23279
	343	1.055151	-11.3518	-373.269	-1.05515
	353	0.876456	-11.1501	-320.539	-0.87646
5 mL/L	323	0.499276	-8.72455	-169.991	-0.49928
	333	0.691742	-9.87754	-240.228	-0.69174
	343	0.61813	-9.86035	-221.879	-0.61813
	353	0.801803	-10.8946	-293.931	-0.8018
7 mL/L	323	0.133313	-5.25641	-48.3165	-0.13331
	333	0.224357	-6.82865	-81.5395	-0.22436
	343	0.249856	-7.33395	-93.0346	-0.24986
	353	0.314169	-8.20522	-119.107	-0.31417

**Table 5:** Activation energies for adsorption of extracted on steel in petroleum medium.

<i>Extract</i>	<i>Conc./ mL/L</i>	<i>E<sub>a</sub><sup>*</sup> / kJ.mol<sup>-1</sup></i>
Petroleum medium	-	10.57
Ficus carica	1	10.23
	3	13.14
	5	1.35
	7	-4.14

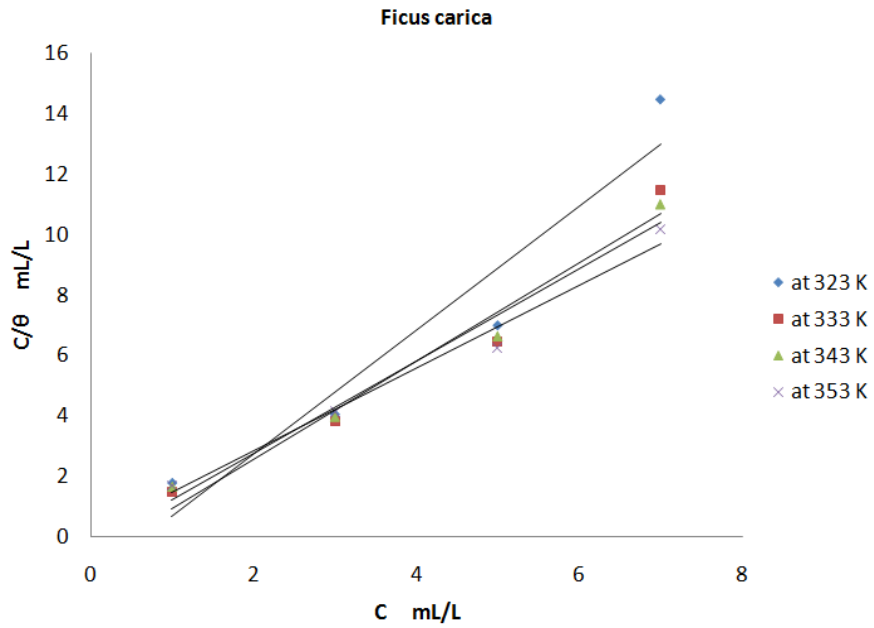


**Figure 1:** Potential – time measurements of carbon steel in petroleum medium in the presence of four concentrations of Ficus carica extract at four temperatures.

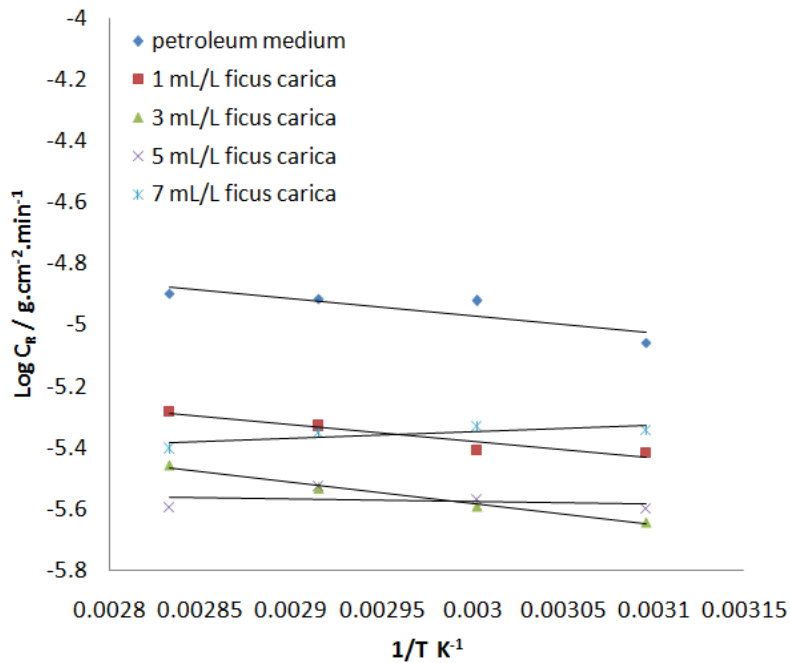


**Figure 2:** Tafel plots of carbon steel in petroleum medium in the presence of four concentrations of Ficus carica extract at four temperatures.





**Figure 3:** Langmuir adsorption plots for steel in petroleum medium with different concentrations of Ficus carica extract at four temperatures.



**Figure 4:** Arrhenius plots of the corrosion rate for steel in petroleum medium in absence and presence of Ficus carica extract.