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Corrosion Inhibition of Rumex Vesicarius Extract on Stainless Steel 304 in Hydrochloric Acid Solution

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Abstract- Stainless steel 304 (SS 304) is corrosion restraint in 2 M HCl by Rumex Vesicarius (RV) extract. This is studied using electrochemical (such as potentiodynamic polarization (PP), electrochemical frequency modulation (EFM) and electrochemical impedance spectroscopy (EIS)) and chemical (mass reduction (MR)) techniques at 25ºC. The extract was adsorbed on surface of SS and is consistent with Langmuir isotherm. The curves from PP indicate that RV extract change the anodic and cathodic Tafel lines. Such exchange led to RV extract play a mixed-kind. The parameters given from thermodynamic were measured from MR at various values of temperatures and were utilized to explain the mechanism of inhibitor. The 304 SS morphology of the coins was tested utilized AFM and ATR-FTIR

Keywords: SS 304, Corrosion inhibition, HCl, MR, PP, EIS, EFM, AFM, ATR-FTIR

I. INTRODUCTION

In human activities utilized many alloy and metals which are liable to various corrosion mechanisms because their disclosing in various aggressive solution or natural to form a constant assembled [1]. Many metal is liable for unique own process of corrosion and it take place a very higher and universally. It is hard for remove completely [2]. Economic reduction in manufactory is the resulted from corrosion problems [3]. Many chemicals hade utilized for protection of alloy surfaces against corrosion by inundation in crucial HCl acid. It is highly utilized in industry in areas such as acidizing oil well, ferrous alloys pickling, , extraction, refinery petroleum, mining and processing manufactory [4]. 304 SS be related to the stainless austenitic steels group [5]. The significant engineering of 304 SS and its assembled material in the world are due to its good mechanical characters [6]. Also 304 SS has utilized in many range of applications, it is highly liable to corrosion because it's thermodynamic instability, in acid solution especially. The research of 304 SS corrosion has requests for heeded of authors to investigate method to particularly lower the corrosion in solution of acid [7]. Rumex vesicarius L., belong the Polygonaceae family, are the presence of the nine define [8]. In Egypt they had been usually utilized as herbs medicinal. vesicarius is an, cooked or fresh eaten and edible weed; traditionally define in as “Humaidah” in Arabic and as “Bladder dock” in English. It is utilized in antimicrobial activities, liver treatment, toothache, problems for digestive, pain, anti-inflammatory and antitumor beside antischistosomal [9]. So to eliminate process of corrosion, corrosion protection is mostly needed. The RV extract is the excellent tests in practice to protect surface of metal from corrosive medium.

The target of study use RV as 304 SS green inhibitor for HCl corrosion.

II.EXPERIMENTAL DETAIL

A. *Metal sample*

The composition of metal sample recorded in Table (1):

Table 1: The wt. % of 304 SS coins was given:

wt%	C	Si	Mn	S	Cr	N	P	Ni	Fe
304	0.08	0.75	2.0	0.03	18-20	0.1	0.045	10.5	the rest

B. *Chemicals*

1) *Inhibitor*

a) *Preparation of plant extract*

Fine powder and Air-dried of RV are liable to extraction with methanol for two days. The container with its contents was stirred and shacked. The RV extract filtered by Buchner funnel and was concentrated with evaporator rotary at bath temperature until reach 40°C to have gummy concentrate extract [10].

ii. Chemical structure of RV

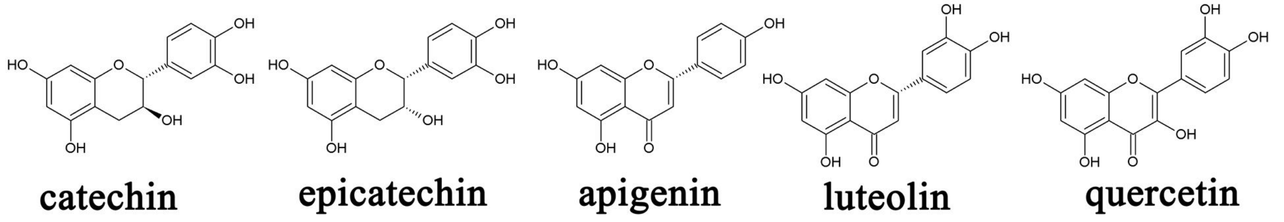


Fig. 1: Chemical markers of *RV*

2) *Solutions:* The electrolyte was a 2 M HCl solution prepared using double distilled water and was standardized by Na_2CO_3. Hydrochloric acid was readying by dilution of HCl 37% (purchased from El-Nasr, Egypt) with double distilled water. The doses range of the used extract were between 50 and 300 ppm.

B. Techniques utilized for corrosion

1) *Mass reduction technique (MR):* For MR tests, square coins of area of surface (20 ml x 20 ml) x 2 which exposed to the corrosive medium were utilized. The coins were scratched with grit papers sizes (300,600 and 2000), clean by acetone. The MR tests were occurred in a bath of thermostat water. The coins were then directly rinsed in the solution test existence and nonexistence various doses of the RV extract. All aggressive medium were air opened. After three hour, the coins were eject, washed, dried, and accurately weighed per thirty minutes. The mean mass reduction for 7 square 304 SS specimens will be given. The protection efficiency (% IE) and (θ) of RV extract for the 304 SS corrosion were measure as next eqn. [11].

$$\% \text{ IE} = \theta \times 100 = [1 - (W/W^o)] \times 100 \qquad (1)$$

Where, W^o and W are the MR, nonexistence and existence adding deferent dose of investigate inhibitor continually.

2) *PP technique:* PP tests were take place in a 3-electrode cell with a Pt electrode counter and a SCE as the electrode reference. The electrode working of 304 SS was cut to sheet fixed in epoxy and the surface flat area was 1.0 cm^2. The working electrode was polisher with papers grit 1200 in size. Before tests, the electrode was inundation in HCl at potential natural for half hours. until arrived to a steady state. The potential was begin from - 500 to + 500 mV vs. OCP (E_{ocp}). All tests were done in freshly readying solutions and outcome data were always recorded at least 3 times to check the validity results. Measured % IE and θ as below [12]

$$\text{IE \%} = 100 \times \theta = 100 \times [1 - (i_{corr(inh)} / i_{corr(free)})] \qquad (2)$$

$i_{corr(inh)}$ and $i_{corr(free)}$ are the current for corrosion in the nonexistence and existence of extract, continually.

3) *EIS technique:* All EIS tests were occurred at E_{ocp} at 25±1°C over a large frequency mean of ($1x10^5$ Hz to x 0.1 Hz). The diameters given of the capacitive loops improve in existence of inhibitors, and are led to the capacitive of the extent of inhibition of process corrosion, contrary to the lower of the C_{dl} which is given as:

$$C_{dl} = 1/(2 \pi f_{max} R_p) \qquad (3)$$

f_{max} = frequency maximum. The IE %and (θ) given from EIS analyses were obtained by the next relation:

$$\text{IE \%} = 100 \times \theta = 100 \times [1 - (R^o_p/R_p)] \qquad (4)$$

R^o_p and R_p are the charge resistance in the nonexistence and existence of extract, respectively.

4) *EFM tests:* EFM tests were done with potential applying signal with 10 mV amplitude with 2 sine waves of 2 and 5 Hz [13]. The maximum peaks were utilized to measure the (CF_2 and CF_3) causality factors, (β_c and β_a) and (i_{corr}) [14]. The inhibition efficiencies % IE_{EFM} was calculated as follows:

$$\%IE_{EFM} = 100 \text{ X } [1 - (i_{corr.} / i^o_{corr.})] \qquad (5)$$

Where, i^{o}_{corr} and i_{corr} are current corrosion in the nonexistence and existence of RV extract, respectively.

All electrochemical tests were occurred utilized Gamry apparatus PCI300/4 Potentiostat/ Zra / Galvanostat; EIS300 utilized for software EIS, and 5.5 Echem Analyst for plotting outcome, value fitting and measurements.

5) *Surface Analysis:* The 304 SS coins utilized for analysis of the 304 SS surface were readying in 2M HCl acid (blank) and with 30 ppm of RV extract at temperature room for one day, first the 304 SS samples had been mechanically scratched with various emery papers up to 2000 grit size. Secondly, the samples gently washed using water bi-distilled, dried carefully and mounted into the performed coins examined by utilized Attenuated total reflection (ATR) with (FTIR) Fourier transform infrared, SEM and AFM spectroscopy used for investigation the film found on the SS surface by Thermo Fisher Nicolet IS10, USA spectral range of 400 – 4000 cm^{-1}.

III. RESULTS AND DISCUSSION

A. *Mass reduction (MR) tests*

The MR of 304 SS coins had been listed after various intervals time of inundation (30, 60, 90, 120, 150 and 180 min) at various temperatures (25-45 °C). Table 2 showed that the % IE values at various temperatures. Existence of RV extract decreases the corrosion rate of the 304 SS pieces in two molar acidic solutions. Rise of %IE observed when the plant extract dose was raised as explained in Table 2. The MR-time curves of 304 SS is shown in Fig (2).

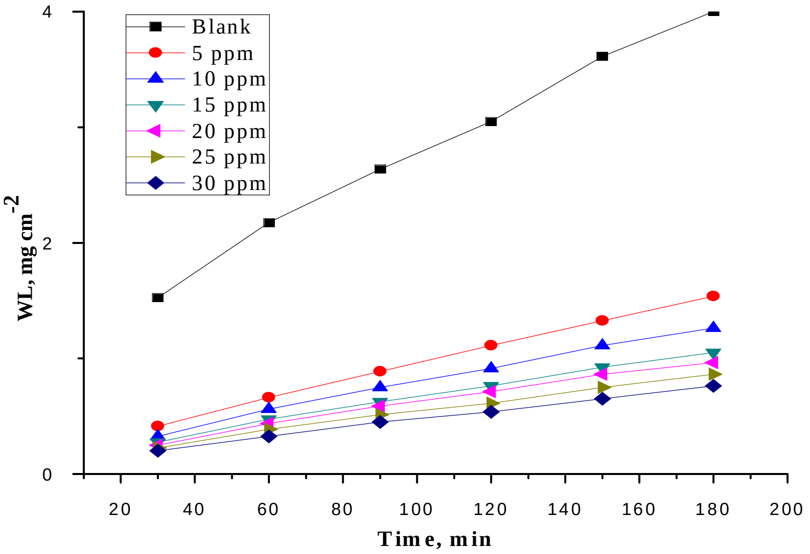


Fig. 2: Curve of time -MR for the 304 SS in the nonexistence and also existence of unlike doses of RV in HCl 2M at 25°C

Table 2: MR measurements at different temperatures at 120 min

Temp. °C	[inh.] ppm	CR mg cm^{-2} min^{-1}	ϴ	% IE
25	Blank	0.025	-	-
	5	0.009	0.635	63.5
	10	0.008	0.701	70.1
	15	0.006	0.750	75.0
	20	0.006	0.766	76.6
	25	0.005	0.799	79.9
	30	0.004	0.824	82.4
30	Blank	0.029	----	----
	5	0.012	0.577	57.7
	10	0.009	0.681	68.1
	15	0.008	0.720	72.0

	20	0.007	0.749	74.9
	25	0.007	0.771	77.1
	30	0.006	0.799	79.9
35	Blank	0.040	----	----
	5	0.018	0.543	54.3
	10	0.014	0.644	64.4
	15	0.012	0.701	70.1
	20	0.011	0.735	73.5
	25	0.010	0.761	76.1
	30	0.009	0.782	78.2
40	Blank	0.041	----	----
	5	0.023	0.446	44.6
	10	0.018	0.554	55.4
	15	0.015	0.635	63.5
	20	0.014	0.671	67.1
	25	0.012	0.701	70.1
	30	0.011	0.739	73.9
45	Blank	0.062	----	----
	5	0.038	0.393	39.3
	10	0.030	0.509	50.9
	15	0.024	0.607	60.7
	20	0.021	0.661	66.1
	25	0.020	0.672	67.2
	30	0.019	0.694	69.4

1) *Adsorption isotherms:* The adsorptive habit of molecules inhibitor are explained by the foundation of adsorption isotherms which can give good thoughts of the kind of the interaction among inhibitor/metal [15].The isotherm give the Fundamental information about reaction among the RV molecules and the surface of 304 SS [16]. The excellent kind of the isotherm habit of the RV extract had been doing by the isotherm Langmuir which is the best among many isotherms. The relationship is linear among (C) and C/Θ (Eq. 6) given in lines straight with the slope = 1 (Fig. 3). K_{ads} is belong to ΔG^0_{ads} as eqn. [17].

$$C/\Theta = 1/K_{ads} + C \qquad (6)$$

$$\Delta G^0_{ads} = RT \ln (K_{ads} \times 55.5) \qquad (7)$$

where K_{ads} = constant equilibrium, R = Universal gas constant, T = temperature in Kelvin and C = dose of RV extract.

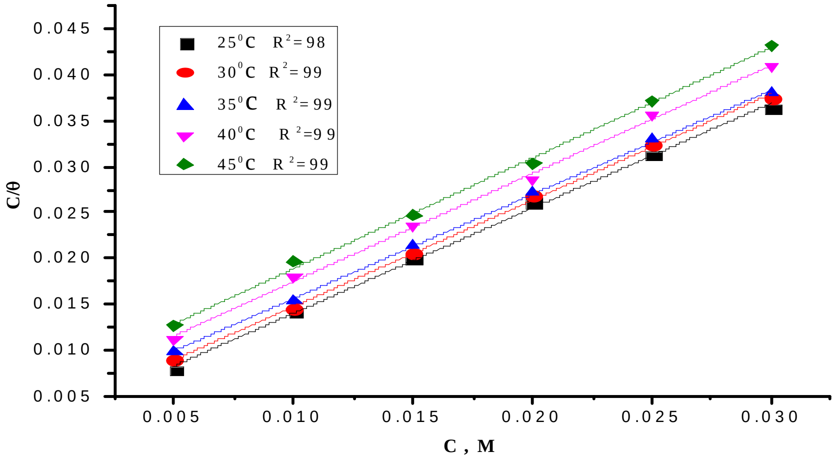


Fig. 3 Plot of Langmuir as C vs. (C/Θ) of RV extract for corrosion of 304 SS

Table 3: Results of 304 SS in RV extract by Langmuir isotherm at various temperatures

Compound	T, K	$k_{ads}x10^{-3}$ M^{-1}	$-\Delta G^{\circ}_{ads}$ kJ mol^{-1}	$-\Delta H^{\circ}_{ads}$ kJ mol^{-1}	$-\Delta S^{\circ}_{ads}$ J mol^{-1} K^{-1}
Rumex	298	383.14	24.7	38.3	50.2
	303	313.48	24.5		49.6
	308	232.56	24.2		50.0
	313	175.13	24.0		50.3
	318	145.77	23.8		49.8

The data of ΔG^{o}_{ads} around 40 kJ mol^{-1} or maximum (chemisorption) include sharing charge or movement from the molecules of inhibitor to the surface of SS to form a coordinate kind bond and a physisorption when data around 20 kJ mol^{-1} or minimum [18]. The measured ΔG^{o}_{ads} data (Table 3) are lower negative than 20 kJ mol^{-1} which given is physisorption and a spontaneous process. RV is in excellent agreement with the ranking of protection efficiency given from various methods. The maximum data of K_{ads} give large extract interaction on the surface of 304 SS [19].

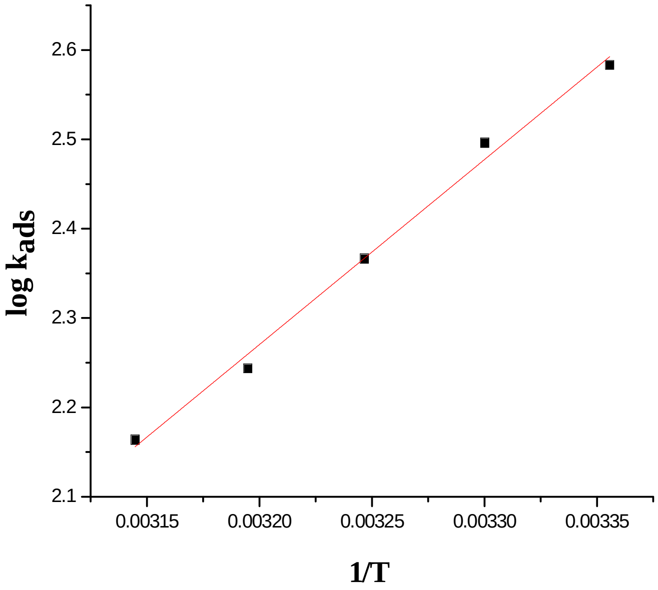


Fig. 4. 1/T vs. log K_{ads} for the adsorption of RV on 304 SS in aggressive medium

2) *Thermodynamic corrosion parameter:* This measured from Arrhenius diagrams from next eqn. (8):

$$k_{corr} = A\ e^{-E^{*}a/RT} \qquad (8)$$

E^{*}_{a} = energy of activation and A = constant pre-exponential Arrhenius. The obtained data of activation energy for 304 SS in existence and nonexistence doses of RV extract are record in (Table 4) which obtained from the slope of 1/T versus log (k_{corr}) curves (Figure 3). The rise in E^{*}_{a} at maximum levels of protection arises due to increase energy barrier [20-22]. The extract components protect both the cathodic and anodic sites on the SS surface. The formulation of transition state equation is illustrated in Eq. (9):

$$k_{corr} = RT/Nh\ e^{(\Delta S^{*}/R)}\ e^{(-\Delta H^{*}/RT)} \qquad (9)$$

Where k_{corr} = rate of metal liquefaction, ΔS^{*} = entropy of activation, h = constant Planck's, and ΔH^{*} = enthalpy of activation. Figure 4 shows plots of 1/T vs. log k_{corr} of RV extract for the SS HCl. Straight lines are given with slopes = (ΔH^{*} /2.303R) and intercepts = [log (R/Nh + ΔS^{*}/2.303R)] , their values are listed in Table 4. The E^{*}_{a} rise with improve inhibitor dose (Table 4) is typical of physical adsorption. The +ve sign of ΔH^{*} given the nature endothermic of 304 SS dissolution process. Data of ΔS^{*} give that the activated complex at the rate determining step favor an association rather than a dissociation, i.e. a disordering break down occurred on going from reactants to the activated state [23] . Also, the data of ΔS^{*} are gradually lower with improving inhibitor doses in all acidic solution.

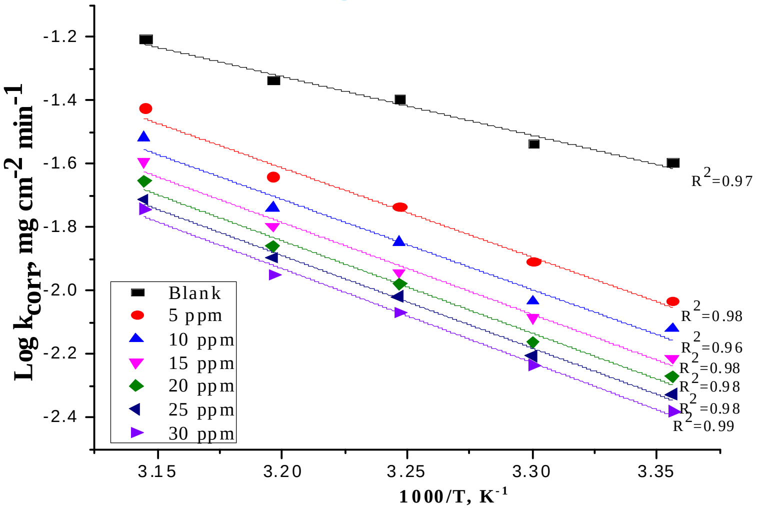


Fig. 5. 1/ T vs.log k_{corr} / T for SS 304 in nonexistence and existence of various doses of RV extract

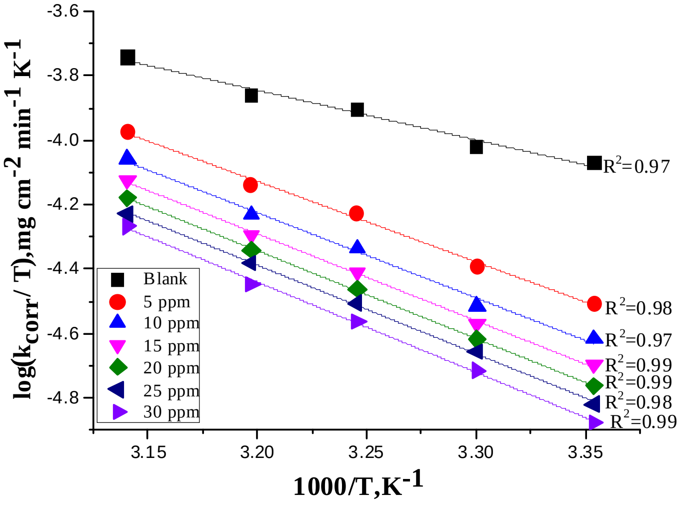


Fig. 6: 1/ T vs. log k_{corr} / T for SS 304 in nonexistence and existence of various doses of RV extract

Table 4 : Parameters for 304 SS in hydrochloric acid in the nonexistence and existence of various doses of RV extract

Conc, ppm	E^*_a kJ mol^{-1}	-ΔH* kJ mol^{-1}	-ΔS* J $mol^{-1}K^{-1}$
Blank	35.4	29.5	176.6
5	53.4	47.9	123.3
10	54.4	50.7	116.2
15	55.3	51.6	114.5
20	55.7	52.3	113.5
25	55.9	53.7	112.9
30	56.7	55.4	110.7

B. EFM measurements

EFM is a corrosion nondestructive test; quickly and directly calculate the data of i_{corr} in the absence Tafel slopes information, and with only a lower signal of polarizing [24]. The higher power of the EFM is the CF which give internal check on the EFM validity for analysis. Figure 7 shows the EFM (i_{corr}.vs F) for RV extract. Maximum peaks were utilized to measure i_{corr}, (β_c and β_a) and CF-2 and CF-3 are listed in Table 6. The outcome data given that the appending of RV at a given dose to the acidic solution lower $i_{corr.}$, led to these assembled protect the 304 SS corrosion in HCl among adsorption. The CF given under various tests are equal approximately to the data obtain from theoretical (2 and 3) led to the calculated value are excellent quality. % IE rise by improving the doses of RV extract and measured by Eq. 10:

$$\%IE = 100 \times [1-(i_{corr}/i^{o}_{corr})] \qquad (10)$$

where i^{o}_{corr} and i_{corr} are current in the nonexistence and existence of RV extract, respectively.

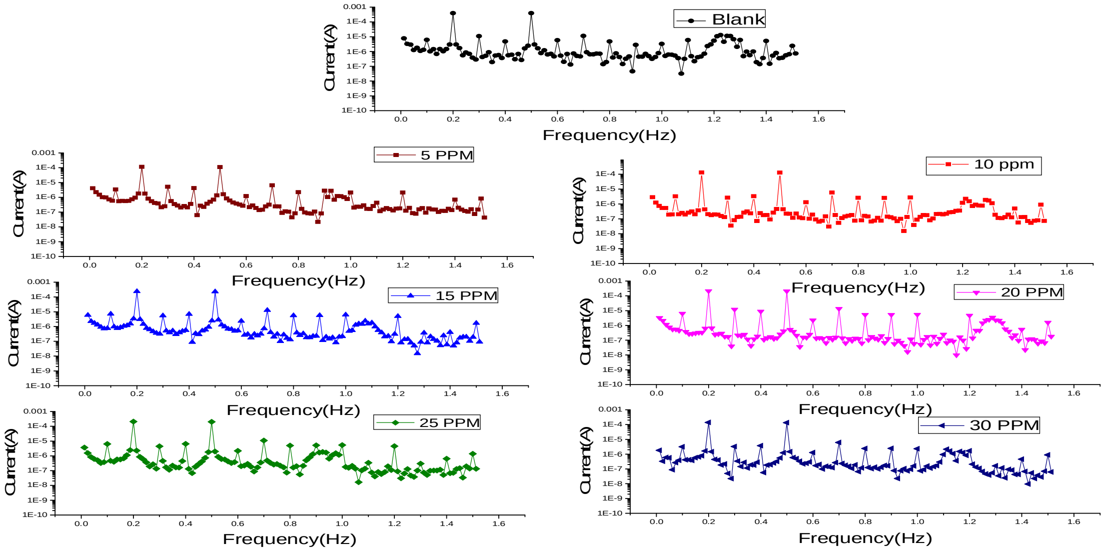


Fig. 7. EFM data for 304 SS in nonexistence and existence of various doses of RV extracts

Table 6: EFM data for the corrosion of 304 SS at various doses of RV extract at 25°C

[inh.] ppm	i_{corr}, μAcm^{-2}	β_c, $mVdec^{-1}$	β_a, $mVdec^{-1}$	CF-2	CF-3	C.R, mpy	Ө	%IE
0.0	627	115	96	1.16	2.75	286	-	-
5	262	80	67	1.73	2.87	120	0.580	58.0
10	238	85	64	1.8	2.79	109	0.619	61.9
15	227	78	66	1.29	2.79	103	0.637	63.7
20	183	96	80	1.56	2.24	83	0.708	70.8
25	169	93	78	1.42	2.19	77	0.729	72.9
30	133	88	68	1.86	2.60	60	0.787	78.7

C. EIS method

The influence of inhibitor dose on the EIS habit of 304 SS in 2 Molar HCl is found in Fig (8, 9). The diagrams show a same kind of Nyquist curves for 304 SS attendance of unlike doses of RV. Attendance of semi-circle show liquefaction of charge transfer process, which have unaffected by attendance of extract [25]. The circuit equivalent model is given in (Fig. 10). It utilized to calculate the EIS value. This includes of the double layer capacitance (C_{dl}), R_s and R_{ct}. (Table 7) illustrated that the data R_{ct} improve and the C_{dl} data break down with raising the inhibitor doses. Due to replacement of water molecules with RV extract on the surface of SS. The maximum values (R_{ct}), are associated generally with lower rate of corrosion [26]. The break down in the Cdl can define from the thickness rise of the electrical double layer or the lower of the dielectric local constant liable that the RV extract molecules by adsorption at interface of the solution / metal [27]. IE given from data of EIS tests are nearly to outcome value from PP tests.

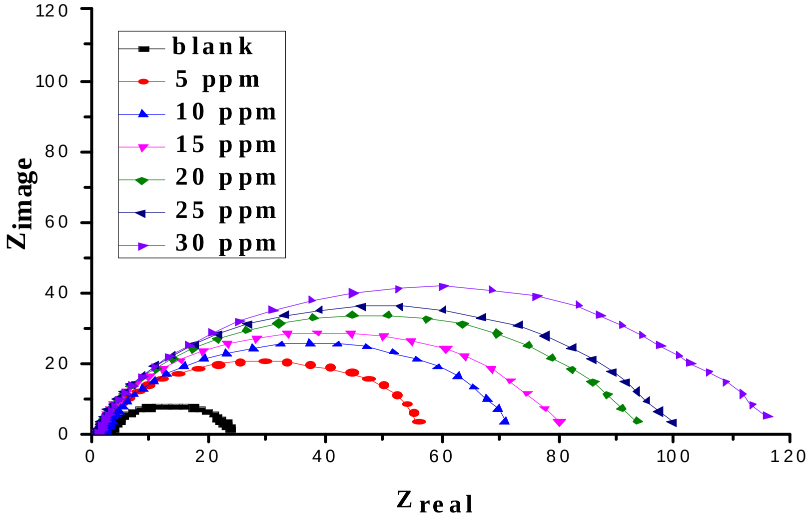


Fig. 8**.** The Nyquist plots for SS 304 in nonexistence and existence of various doses of RV extract

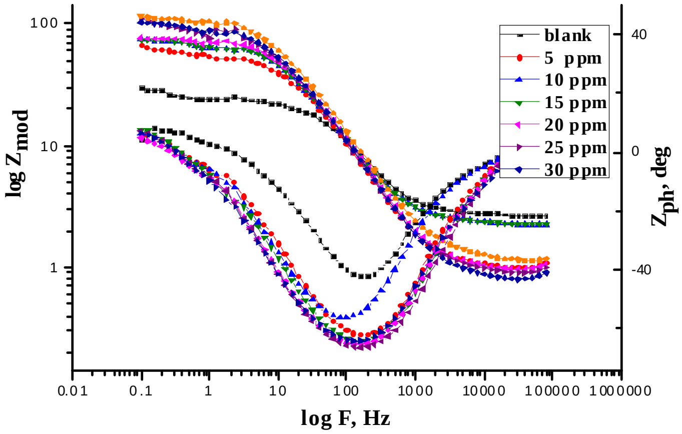


Fig. 9**.** The Bode plots for 304 SS in nonexistence and existence of various doses of RV extract at 25°C

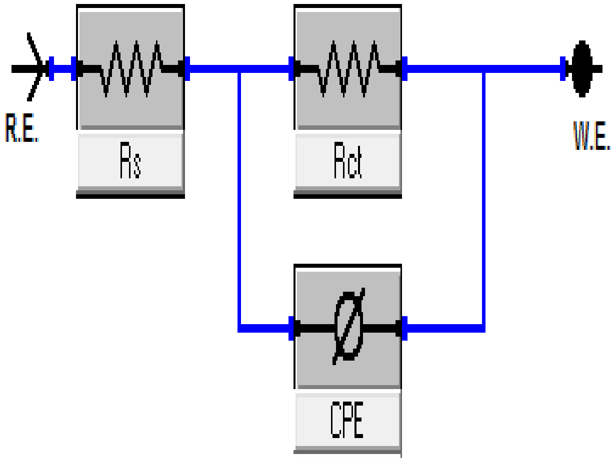


Fig. 10**.** Equivalent circuit utilized to fit the EIS results

Table 7. EIS value of SS 304 in nonexistence and existence of various doses of RV extract at 25ºC

[inh.] ppm	R_{ct}, Ω cm^{-2}	C_{dl}, µFcm^{-2}	θ	%IE
0.0	20	878	----	----
5	53	135	0.623	62.3
10	64	95	0.687	68.7
15	69	75	0.716	71.6
20	84	54	0.762	76.2
25	88	48	0.772	77.2
30	101	39	0.802	80.2

D. PP tests

Figure 11 give the TP diagrams for 304 SS in acidic solution in the nonexistence and existence of doses of RV at 25ºC. The outcome data from Figure 11 given that both anodic SS liquefaction and reduction cathodic hydrogen were protecting by RV extract were appending to 2 Molar acid and this pronounced protection with improving RV extract dose. Tafel lines are change to more potential

–ve and +ve with diagrams of blank by raising the dose of RV extract. This habit led to RV extracts play as physical protection [28]. Appending of RV are exchange the E_{corr} data to -ve direction. The classification of inhibitor as a cathodic or anodic kind when E_{corr} higher than 85 mV [29]. The outcome data give the rise in extract dose, lower the i_{corr}, but the (β_a, β_c)are approximately constant and parallel led to the retardation of the 2 reactions were influence without exchanging the liquefaction mechanism[30].

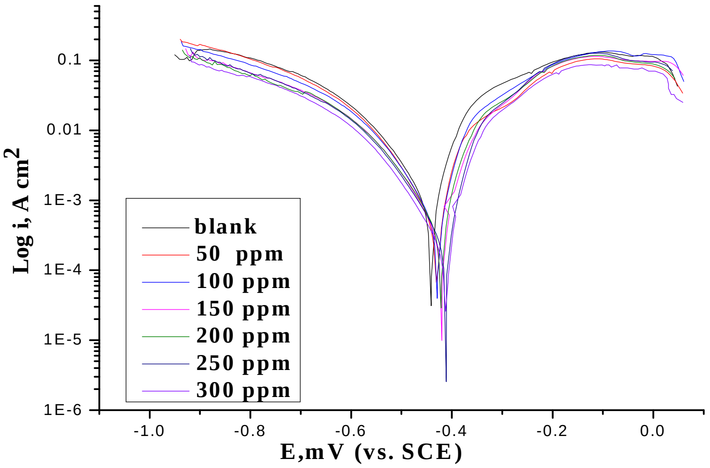


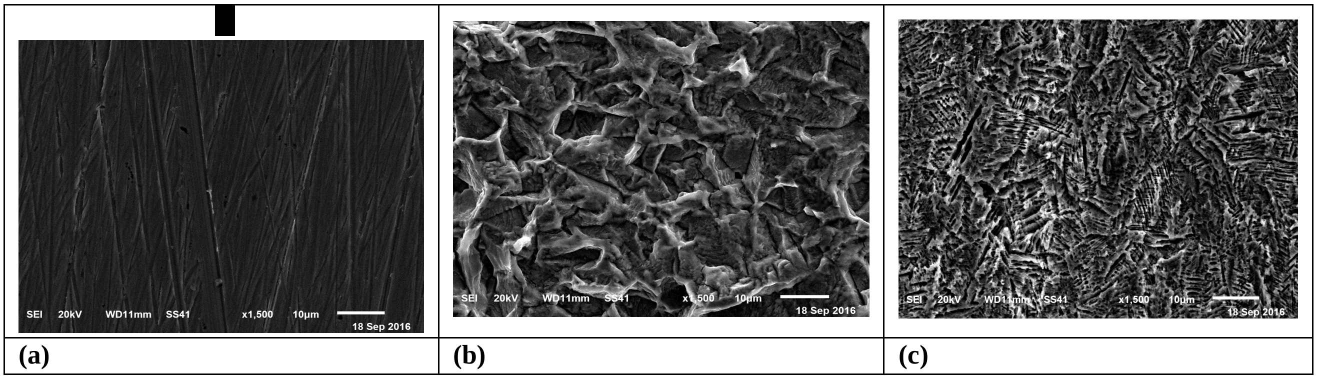
Fig. 11. PP diagrams for the dissolution of SS 304 in the nonexistence and existence of various doses of RV extract

Table 8: PP parameters of 304 SS including various doses of RV extract

[inh.] ppm	- E_{corr}, mV (vs.SCE)	i_{corr}, µA cm^{-2}	β_c mV dec^{-1}	β_a mV dec^{-1}	C.R, mpy	Ɵ	% IE
0	441	997	107	51	455	----	----
5	430	576	100	48	263	0.424	42.4
10	429	531	98	41	242	0.467	46.7
15	420	480	116	56	219	0.518	51.8
20	421	438	107	41	200	0.561	56.1
25	411	309	106	41	141	0.690	69.0
30	411	246	107	43	112	0.753	75.3

E. Surface characterization

1) *SEM test*: SEM micrograms of polished 304 SS without immersion and exposed for 1 day in 2 M HCl solutions in nonexistence and existence of 30 ppm RV extract were shown in Figures12a-c respectively.Figure12a SEM spectroscopy gives the image of polished 304 SS surface. When RV extract nonexistence (Fig. 12b) the outcomes displayed that the entire surface of 304 SS covered with dense pored layer of corrosion product (oxide film); sever destruction occur on the surface and cracks was observed, there for the specimen surface could not be seen. (Figure 12c) showed the image of the specimen's surface when 30 ppm RV extract was present. The specimen surface is nearly not having destruction and it is smooth, this referred to the presence of an effective protective layer on the surface of SS 304, furthermore assure the elevated inhibition efficiency of RV.



(a) **(b)** **(c)**

Fig. 12a-c: SEM micrographs of 304 SS surface (a) free surface of 304 SS, (b) after 1 day of rinsed in 2 M HCl and (c) after 24 hours of rinsed in acid + 30 ppm RV

2) *AFM study:* The roughness of SS 304 surface by utilized RV extract were given in nonexistence and existence RV (30 ppm) was tested by AFM maximum-resolution; the outcome data are shown in Figs.13-15, continually. The outcome data from the both nonexistence and existence RV, the roughness of surface (R_{max}) data = 501.9 nm, 40.02 nm and 241.07nm, continually. The data of R_{max} lower with the appending of RV to the medium, led to that the film covered deposited in the presence of RV prevent a smooth and shiny surface [31]. Our analysis revealed that a deposit with a smooth surface, was given when RV (30 ppm) was appending to the electrolyte.

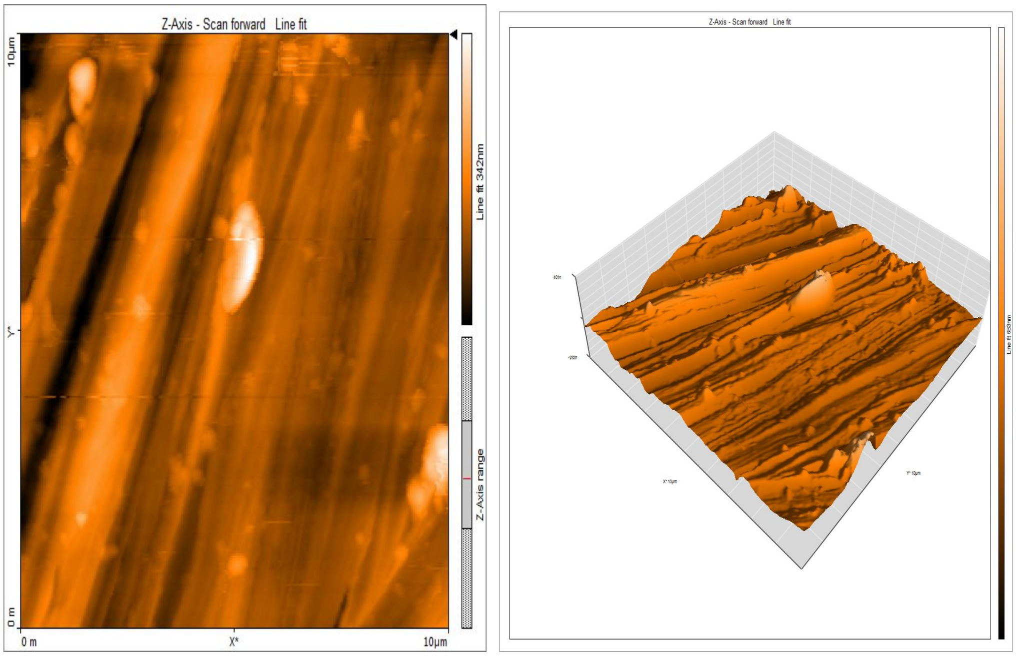


Fig. 13. AFM 2D (a) and 3D (b) of 304 SS free surface

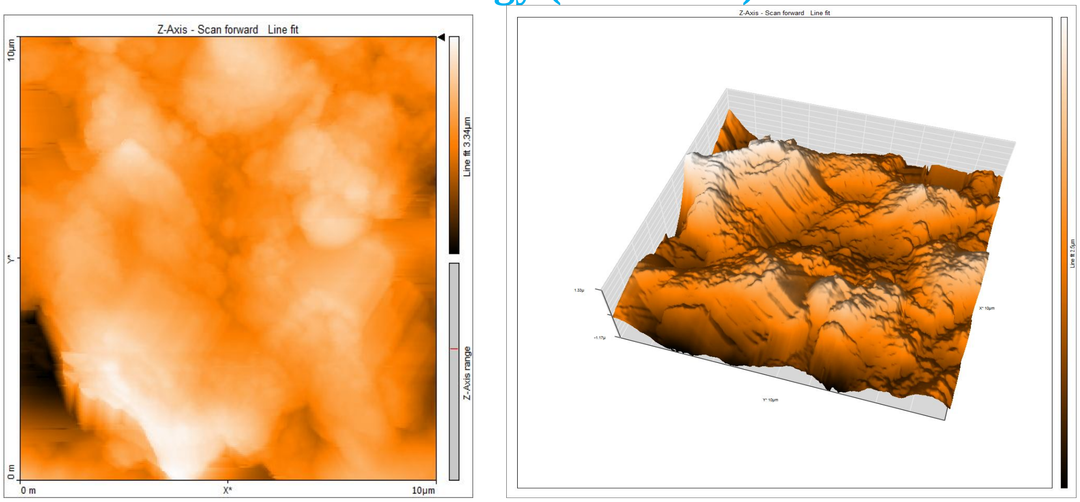


Fig. 14. AFM 2D (a) and 3D (b) of 304 SS electrodeposits in 2M HCl

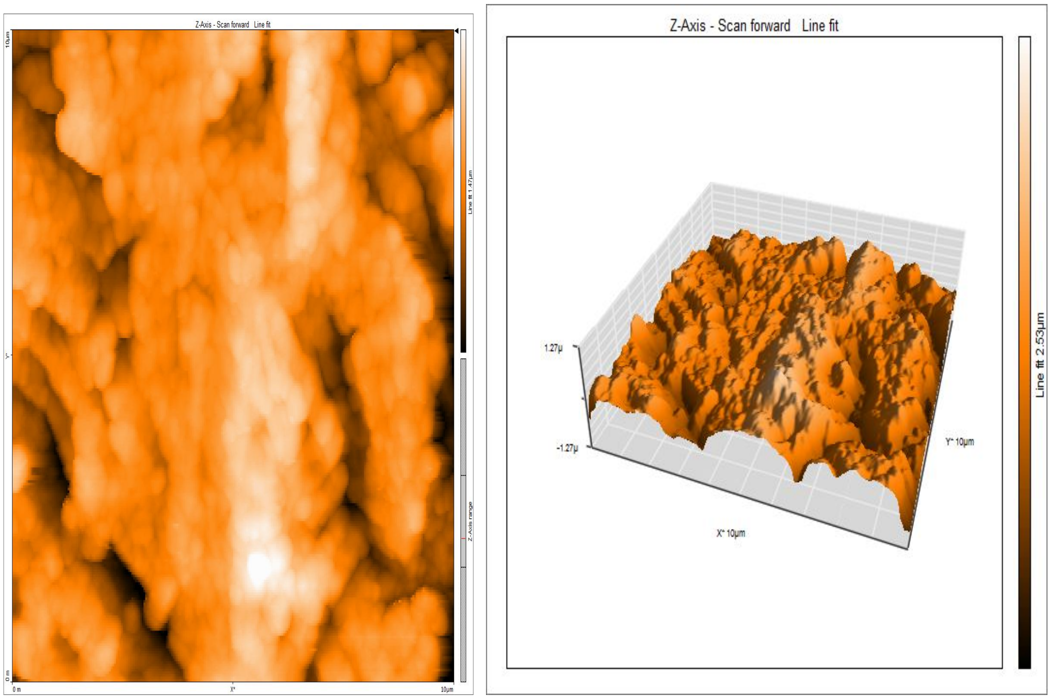


Fig. 15. AFM images 2D (a) and 3D (b) of SS electrodeposits in presence of RV extract

3) *ATR-FTIR Analysis:* Organic and inorganic samples had been detected by ATR- FT-IR which do quantitative and qualitative analysis. Chemical bonds in a molecule had been recognized by generate an infrared absorption spectrum .Functional groups and characterizing covalent bonding information had been detected by FT-IR which is influential analytical device [32]. ATR-FT-IR spectrum of the corrosion product at 304 SS surface in 2M HCl does not show any useful adsorption peaks; therefore, the corrosion product of 304 SS is not IR active. The finger print spectra of the stock RV extract and the 304 SS surface after immersion in 2M HCl + 30 ppm of RV extract for 6 hours was obtained and compared to each other it was obviously clear that the same finger print of RV stock solution present on 304 SS surface except the absence of some functional group and it suggested to be due to reaction with HCl. From Figure 16 there are small shift in the peaks at 304 SS surface from the original peak of the stock inhibitor solution, these shifts indicate that there is interaction between 304 SS and some of the inhibitor's molecules.

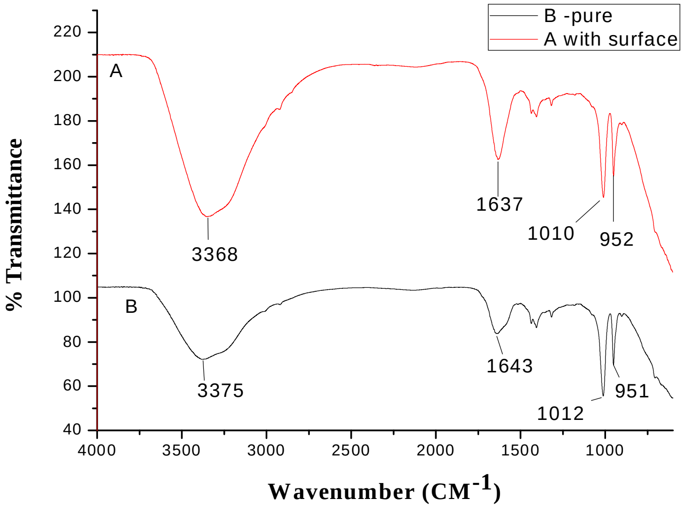


Fig. 16. ATR- FTIR finger print spectra of RV stock solution (1000 ppm) (B) and adsorbed layer of RV on 304 SS surface (A)

F. Corrosion inhibition mechanism

RV extract adsorption is illustrated upon the basis that the inhibitor adsorption carried by hetero atoms (viz., N) which are in different constituents of extract beside the availability of π- electrons in the aromatic system [33]. The RV extract phytoconstituents contain many alkaloids like piperundecalidine, piperine, piperlongumine, piplartine, pipernonaline, and piperlonguminine etc., a few hitherto unidentified steroids and their glycosides and some lowering sugars [34]. Many active centers are present in phytochemical constituents of RV extract like sulfur, oxygen and nitrogen, which are considered as centers of adsorption. Temperatures study results showed that the mechanical adsorption happen through the physical adsorption, this impact of the extract cannot be belonged to a specific component.

IV. CONCLUSIONS

In view of the above outcomes, the next conclusions are given below:

The RV extract gives great execution as corrosion protection for 304 SS in hydrochloric acid. the RV extract outcomes got from all techniques demonstrated that the inhibiting action rises when the extract dose rises and decreases with the rising the solution temperature. When the plant extract is added the double layer capacitances reduce and charge travel resistance rises regardless of blank solution. The above conclusion confirms the adsorbed RV extract on surface of 304 SS. The corrosion protection of the RV extract had been adsorbed on the surface of 304 SS belong to isotherm Langmuir. TP outcomes show that these extract ac as mixed-kind inhibitor. Techniques which are used to detect the inhibition abilities are MR, PP, EFM and EIS are in reasonable good agreement.

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