## **Corrosion Inhibition of Carbon Steel in 2.0M HCl Solution Using Novel Extract (***Pulicaria undulate***)**

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**Abstract:** Plant *Pulicaria undulate* (PU) extract is examined as a corrosion inhibitor for carbon steel (CS) dipped in 2.0M HCl. The study was conducted utilizing ac impedance spectroscopy (EIS), Mass loss (ML), polarization (PDP) tests, and surface checks were utilized to illustrate the importance of this PU extract to prevent the corrosion process for CS. The influence of temperature and concentration of PU on the efficacy of inhibition were tested. The corrosion mechanism occurs when the PU extract molecules block the active center in the electrode surface. Langmuir isotherm is the isotherm that is applied in the process of adsorption. The effect of temperature at various temperatures on the corrosion efficacy was investigated in case of the presence and absence of the extract. Finally, thermodynamic parameters for the activation and adsorption processes were determined. Results of all methods used were in good agreement.

#### Keywords: Corrosion; CS; PU; EIS; PDP; EFM.

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#### 1. Introduction

Corrosion is a naturally occurring process that affects our life and causes degradation to domestic gadgets, automobiles, airplanes, public roads, bridges, and distribution systems [1]. The main reason for the corrosion of metals is their tendency to reach a stable state. Several metal alloys are unstable and need to interact with the surrounding atmosphere to obtain minor energy by forming metal complexes [2]. CS is used in many industries such as refineries of oil and gas, bridges, and machines. Acidic media such as hydrochloric acid is used in the study of carbon steel decomposition due to acids are usually utilized materials for pickling of metals, cleaning boilers, acidizing oil wells, and recovery of ion exchangers. In addition to the previous, HCl is one of the acids that are widely used in improving the productivity (stimulation) of wells in oil and gas production, and over several years is utilized to raise the primary productivity of new wells as well as restore the productivity of old wells [3]. Acids enhance the metal dissolution rate and are responsible for material damage. To control the process of metallic corrosion, there are several tests, but one of the most significant practical tests used is utilizing corrosion inhibitors in the industrial field and academic studies and inhibit in the literary survey [4]. Corrosion inhibitors are those substances that, if added in lesser amounts to corrosion solutions, may lower or prevent the interaction of the metals with these environments. Inhibitors are those substances that, if added in lesser amounts to corrosion solutions, may lower or prevent

the interaction of the metals with these environments. Organic compounds that contain heteroatoms such as nitrogen, oxygen, and sulfur atoms, in addition to the many other linkages that let them adsorb on the metal's surface and protect it from corrosion [5]. Corrosion inhibitors found in nature are environmentally beneficial, readily available, and inexpensive, with renewable sources of supply [6–16]. We investigated environmentally friendly materials (PU) extract as CS corrosion inhibitors in a 1M HCl solution in this study, and we chose PU because of its properties: it contains many organic compounds that are ideal in the process of protecting metals, as well as its affordability and availability. The percentage protection productivity was calculated using three techniques: mass loss, electrochemical tests, and a combination of the two. The effect of higher temperatures on corrosion rate was measured and demonstrated.

### 2. Materials and Methods

### 2.1. Materials and reagent.

By diluting a reagent of analytical grade HCl 37 percent with bi-distilled water, an acid corrosive environment (1 M HCl) was created. C (0.2 percent), Mn (0.35 percent), P (0.024 percent), Si (0.003 percent), and balance Fe make up the composition of the applied CS in weight percent. For the ML tests, a 0.2 cm thick CS sheet was mechanically press-cut into 22 centimeter coupons. The samples were abraded with 400 and 2000 grade emery, cleaned and washed with acetone, sunk with bi-distilled water, and dried with filter paper. After that, the coupons were ready to be used in the corrosion tests. Using bi-distilled water, proper doses of the acid were created. In 2.0M HCl, PU extract dosages range from 50 to 250 ppm.2.2. Preparation of plant extract and chemical structures of main constituents.

#### 2.2. Preparation of plant extract and Chemical structures of main constituents.

Fine-powdered, air-dried whole plant of PU (1.2 kg) was extracted under reflux three times with 70% methanol/ water and then evaporated under reduced pressure and temperature. The purification was achieved on a Sephadex column (35 cm 1.5 cm) column using methanol as an eluent [17,18]. The main components found in the methanolic extract are 1) 5,7,2,3,4 Penta hydroxyl isoflavone-4'-O-b-glucopyranoside; 2) kaempferol; 3-kaempferol 3) O-b-glucoside; 4) quercetin; 5) quercetin3-O-b-glucoside; 6) quercetin 3-O-b-galactoside; 7) quercetin 3,7-di OCH<sub>3</sub>; 8) caffeic acid.

## 2.3. Mass loss (ML) tests.

ML was achieved on CS coins with a cubic form (2cm×2cm×0.03 cm) in 2.0 M HCl presence and absence of altered doses of PU. Every sample was weighed by an electronic balance and then immersion in the acid environment (50 ml). The dipping time was 120 min at the range of temperature from 303 to 333 K. After dipping, the CS specimen was cleaned by alcohol washing, and the sample was massed again to measure the dissolution rate [19].

## 2.4. Electrochemical tests.

The CS was utilized for electrochemical tests (potentiodynamic polarization (PDP) and AC impedance (EIS) procedures). Prior to each investigation, the surface of CS coins was mechanically scratched with altered grades of emery papers and rinsed with bi-distilled water. Electrochemical techniques were carried out by utilizing Potentiostat/Galvanostat (PCI4-G750)

with software DC 105, EIS 300 for measurements, linked to a computer for data documented and saved. Each experiment was achieved on a newly abraded electrode utilizing a freshly prepared electrolyte. PDP technique was utilized through a cell involving three classic electrodes, which is the working electrode that is utilized CS for study, the second electrode is the reference electrode, and the third electrode is a platinum disk ( $\emptyset$ 1mm, 10 mm). The samples were abraded with 400 and 2000 grade emery, cleaned and rinsed in acetone, buried in bidistilled water, and dried on filter paper. The coupons were then ready for use in the corrosion tests. Proper dosages of the acid were generated using bi-distilled water. PU extract doses in 2.0M HCl vary from 50 to 250 ppm. All potentials were measured with the SCE in mind [20, 21]. The experiment was carried out with AC signals ranging from (100 kHz to x 0.1 Hz) and peak amplitudes of 10 mV at OCP. Using the Gamry Echem program and the charge transfer resistance to know the quality of protection, all of the impedance results were compatible with the suitable equivalent circuit.

2.5. Morphology of the surface.

2.5.1. Atomic force microscopy (AFM).

AFM adapted tests provide data on the surface examination of CS samples with metric linear purity. Measured Knowledge is implemented and valued through persecution From the SPM management computer code [22].

2.5.2. Attenuated Total Reflection Infra-Red (ATR-IR).

ATR-IR is the performance by utilizing Thermo Fisher Nicolet IS10, USA spectrophotometer [23].

#### 3. Results and Discussion

#### 3.1. Mass loss (ML) method.

The ML data of CS in 2.0M HCl in attendance and the absence of altered doses of PU were achieved in Fig. 1 and are summarized in Table 1. The  $\eta$ % was measured by utilizing the next Eq. [24,25]:

$$\eta\% = \frac{CR_{corr} - CR_{corr(inh)}}{CR_{corr}} \times 100$$
(1)

where  $CR_{corr}$  and  $CR_{corr(inh)}$  are the rates of CS sheet corrosion in presence and absence of altered doses of extract, correspondingly. Table 1 displays that the utilized extract hindrance the CR of CS in 2.0M HCl at all doses under research. The CR is found to rely on the dose of the extract.  $\eta$ % improves with raising in the dose of PU [26].

**Table 1.** List of corrosion rate values at 120-minute immersion at different temperatures for CS in 2.0M HCl in without and with of different doses of PU.

[inh.]	$CR_{corr} (mg cm^{-2} min^{-1})$							
ppm	25 °C	30 °C	35 °C	40 °C	45°C			
Blank	0.013	0.02	0.033	0.048	0.085			
50	0.0067	0.0102	0.0157	0.0212	0.034			
100	0.005	0.007	0.011	0.014	0.023			
150	0.0037	0.0057	0.0088	0.0096	0.0152			
200	0.002	0.003	0.004	0.006	0.007			
250	0.002	0.0028	0.0041	0.0051	0.0065			

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Figure 1. Plots of Mass loss vs. time for CS in 2.0M HCl in case of the free acid and in the presence of various doses of PU extract at 25°C.

#### 3.2. Effect of temperature.

The results obtained from Table 2 showed that the  $\eta$ % are initiated to improve with raising the temperature from 25°C to 45°C. This performance can be interpreted because the adsorption of PU molecules on the surface of CS is chemisorption [27].

[inh.]			ղ%		
ppm	25 °C	30 °C	35 °C	40 °C	45 °C
50	47.54	49.51	52.59	56.32	60.01
100	61.12	63.61	65.63	70.82	73.05
150	70.73	71.63	73.51	80.16	82.06
200	82.51	85.09	86.68	88.28	91.77
250	84.26	86.38	87.72	89.52	92.34

 $\label{eq:table 2. List of $\eta\%$ values at 120-minute immersion at different temperatures for C-S in 2.0M HCl attendance and absence of altered dose of PU.$ 

The ( $E_a^*$ ) of the dissolution process was calculated using Arrhenius eq. (2) [28]:  $k_{corr} = A \exp(-E_a^* / RT)$  (2)

Figure 2. refers to the straight lines given by a plot [logk<sub>corr</sub> vs.1/T], which can calculate the  $E_a^*$  from their slopes which are illustrated in Table 3.  $\Delta$  H<sup>\*</sup>and  $\Delta$ S<sup>\*</sup> are measured from transition state theory utilizing eq. (3) [29]:

 $k_{corr} = RT/Nh \exp \left(\Delta S^*/R\right) \exp \left(-\Delta H^*/RT\right)$ (3)

where  $\Delta S^*$  and  $\Delta H^*$  are entropy and activation enthalpy. Plot  $[\log k_{corr} / T \text{ vs. } 1/T]$  provided straight lines Figure 3. from their slopes  $\Delta H^*$  and their intercepts  $\Delta S^*$  can be calculated and reported in Table 3. The outcome data dictate that  $E_a^*$  decreases in the existence of PU extract than in its nonexistence, demonstrating chemical adsorption, the importance that the block of CS surface activities by bonding and hindering. Positive  $\Delta H^*$  data designate the endothermic activation procedure [30].



Figure 2. Plotting log CR against 1/T for CS in the attendance and absence of various concentrations of PU.



Figure 3. Plotting log CR/T against 1/T for CS in the attendance and absence of various concentrations of PU.

<b>Table 3.</b> Activation parameters ( $E_a^*$ , $\Delta H^*$ , and $\Delta S^*$ ) for CS in the attendance and absence of various	us
concentrations of <i>PU</i> .	

Cinh, ppm	Ea <sup>*</sup> , kJ /mol	$\Delta \mathbf{H}^*$ , kJ /mol	- $\Delta S^*$ , J /mol.K
Blank	72.39	71.13	42.51
50	63.12	60.76	82.56
100	59.11	56.30	100.10
150	54.33	51.69	117.68
200	50.99	47.71	136.10
250	46.85	44.17	148.12

#### 3.3. Adsorption isotherms.

The best isotherm among the experimental data and the isothermal functions was achieved in the range temperature studied utilizing the Langmuir isotherm adsorption [31], known by the next relation:

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$$\frac{c}{\Theta} = \frac{1}{K_{ads}} + C$$

 $K_{ads}$  is the adsorption equilibrium constant, and (C) is the extract's doses. For PU the linear regressions between C/ $\theta$  and C are shown in Figure 4. The results showed that all linear lines and slopes are near 1 and designate that PU molecules were adsorbed in 2.0M HCl on the CS surface. The  $K_{ads}$  could be determined from the intercepts of lines on the C /  $\theta$  axis, and  $K_{ads}$  was related to the standard free adsorption energy,  $\Delta G^o{}_{ads}$  as follows:

$$\Delta G_{ads}^o = -RTln(55.5K_{ads}) \tag{5}$$

The  $\Delta G^{o}_{ads}$  data at all temperatures are recognized in Table 3. The ( $\Delta H^{o}_{ads.}$ ) was measure agreeing to the Van't Hoff eqn.

$$\log K_{ads} = -\Delta H^{o}_{ads}/2.303RT + constant$$
(6)

Plotting (log K<sub>ads</sub>) vs. (1/T) gives a straight line as displayed in Figure 5, the slope = (- $\Delta H^{o}_{ads}/2.303R$ ), from this slope; the  $\Delta H^{o}_{ads}$  data was measured and is recorded in Table 4. Then by applying the next balance:



$$\Delta G^{o}_{ads} = \Delta H^{o}_{ads} - T\Delta S^{o}_{ads}$$
(7)

Figure 4. Langmuir plots for CS in 2.0M HCl, inclosing altered doses of PU at various temperatures (25 - 45°C).





Tuble 4. Earginan parameters of r e extract for es in 2.000 mer obtained at artered temperatures.								
Temp. °C	K <sub>ads</sub> x 10 <sup>-3</sup> M <sup>-1</sup>	$\mathbb{R}^2$	-ΔG° <sub>ads</sub> kJ mol <sup>-1</sup>	-∆H° <sub>ads</sub> kJ mol <sup>-1</sup>	-ΔS° <sub>ads</sub> kJ mol K <sup>-1</sup>			
298	15.46	0.996	16.74	19.66	122.16			
303	16.63	0.997	17.20		121.68			
308	17.83	0.999	17.66		121.20			
313	22.71	0.999	18.58		122.19			
318	24.52	0.999	19.08		121.84			

**Table 4.** Langmuir parameters of PU extract for CS in 2.0M HCl obtained at altered temperatures.

The data of Table 4 confirm the spontaneous adsorption of PU extricate on the Through the negative data, CS surface obtained  $\Delta G^0_{ads}$ , whose negative value (spontaneous), when negative values are lower than 20 kJ /mol, adsorption is physical adsorption. The exothermic process can relate to either physical or chemical adsorption. However, the kind of adsorption is determined by the value. The estimated values of S<sup>o</sup><sub>ads</sub> showed that the adsorption process was accompanied by a drop in entropy [32].

3.5. Electrochemical measurements.

#### 3.5.1. PDP.

In which an electrochemical solution's potential is formed and  $E_{corr}$  is measured, and  $I_{corr}$  from the Tafel curve ( $I^{o}_{corr}$ ) slope is estimated, which is determined by the following equation [33]:

$$\eta_{PP}\% = \theta x 100 = \left[\frac{I_{corr}^o - I_{corr}}{I_{corr}^o}\right] x 100$$
(8)

where  $I_{corr}^{o}$  and  $I_{corr}$  are the current absence and attendance of PU, correspondingly. PDP bends for CS in 2.0M HCl, the existence and absence of PU dose are displayed in Figure 6. The parallel Tafel lines  $\beta_{a} \& \beta_{c}$  indicate that both anodic and cathodic mechanisms do not impact by adding the PU to the corrosive environment. The data in Table 5 displays that the lower  $I_{corr}$  data in the existence of PU without affecting important exchanges in  $E_{corr}$  recommends that the PU is mixed kind [34]. In all doses,  $\beta_{c}$  is larger than  $\beta_{a}$ , signifying that although the protection is under mixed control, the influence of PU on the cathodic is more distinct than anodic polarization.



Figure 6. PDP bends for dissolution of CS presence and absence of altered doses of PU at 25°C.

	<b>Fuble C.</b> Effect of the extract dobe on the dissolution of CD in 2.011 field at 25 C.								
Conc. (ppm)	- E <sub>corr</sub> , mV vs SCE	I <sub>corr</sub> , mA cm <sup>-2</sup>	β <sub>c</sub> mV dec <sup>-1</sup>	β <sub>a</sub> mV dec <sup>-1</sup>	C.R mpy	θ	η%		
Blank	470	522	111	72	238				
50	462	271	101	65	124	0.481	48.1		
100	458	172	98	60	78	0.670	67.0		
150	450	158	103	69	72	0.697	69.7		
200	458	110	97	68	51	0.789	78.9		
250	454	86	95	71	39	0.835	83.5		

Table 5. Effect of PU extract dose on the dissolution of CS in 2.0M HCl at 25°C.

3.5.2. EIS results.

Figure 7 displays the Nyquist and Bode diagrams for CS surface dipping at 2.0M HCl absence and existence of different PU doses at 25°C. As the PU dose decrease, the semicircle's diameter rises [35], by using the charge transfer resistance as a function of knowing the quality of protection by the next balance.

$$\eta_{EIS}\% = \theta x 100 = \left[ \left( R_{ct} - \frac{R_{ct}^0}{R_{ct}} \right) x 100 \right]$$
(9)

 $R_{ct}$  and  $R^{\circ}_{ct}$  are the resistances in existence and absence PU, correspondingly.



Figure 7. EIS Nyquist (a) and Bode (b) bends of CS in 2.0M HCl solutions with and without of altered doses of PU at 25°C.

The matching circuit that defines CS alloy and electrolyte is established in Figure 8. Table 6 reported the EIS parameters. The deviation from the ideal semicircle was commonly due to the dispersion of frequencies and to the surface's inhomogeneity, grain boundaries, and impurities. Increasing the doses of the PU raises the charge transfer resistance ( $R_{ct}$ ) due to the increase in the thickness of the adsorbed layer and decreases the double-layer capacitance ( $C_{dl}$ ), due to the rise of the thickness of the electrical double layer or/and the lesser in the dielectric constant due to the replacement of the adsorbed water molecules on CS surface by the extract molecules", recommends that the PU molecules function by adsorption at the CS /interface [36].



Figure 8. The employed equivalent circuit for appropriate the EIS values.

Table 6.	EIS for the	dissolution o	f CS in	2.0M HCl	attendance an	nd absence of	altered	doses of PU	f at 25 °C.

Conc. (ppm)	Y <sub>0</sub> , (μΩ <sup>-1</sup> s <sup>n</sup> cm <sup>-2</sup> ) x10 <sup>-6</sup>	n	$R_{ct}, \Omega cm^2$	С <sub>dl</sub> , µF ст <sup>-2</sup> x10 <sup>-6</sup>	θ	η%
Blank	215	0.857	36.5	115		
50	246	0.34	50.8	103	0.281	28.1
100	244	0.817	69.8	98	0.477	47.7
150	233	0.810	88.1	94	0.586	58.6
200	231	0.775	136.1	85	0.732	73.2
250	228	0.737	194.3	75	0.812	81.2

3.6. Morphology of surface.

3.6.1. Attenuated total reflection infra-red (ATR-IR) analysis.

An FT-IR spectrophotometer is a useful tool for determining the presence of functional groups in PU and the interaction between functional groups and metal surfaces [37]. Figure 9 shows broad peaks of PU and PU with CS. After corrosion, there are some peaks displacement between the spectra of the PU and the adsorbed extract from the CS surface, and a few peaks disappear or become less noticeable. This implies that PU interacts with CS through the functional groups found in PU molecules, resulting in corrosion prevention.



Figure 9. (a) CS ATR-FTIR after dipping in 2.0M HCl + 300 ppm of PU; (b) PU before immersion.

3.6.2. Atomic force microscopy (AFM) analysis.

The AFM is the most appropriate method for measuring surface topography as it can provide three-dimensional (3D); AFM considered the surface image details at the nano-to micro-scale to research the degree of inhibition in the test solution for dissolution of CS interfacing [38]. In the absence and existence of PU, Figure 10 displays the three dimensions (3D) using AFM morphologies for CS of 2.0M HCl. Root Mean roughness (Sq) square and mean roughness (Sa). AFM picture in 2.0M HCl shows CS surface corrosion with hard degradation but good quality in the presence of PU. The values (Sa) and (Sq) in Table 7 show that the surface of the CS is smoothed by the adsorbed surface layers of the PU on the CS [39].



A) Blank B) With 300 ppm of PU compound Figure 10. Three-dimension AFM images for CS surface (A) Blank (CS with 2.0M HCl only); (B) CS immersed in 2.0M HCl containing 300 ppm of PU.

Specimen	Average roughness (Sa) [nm]	RMS roughness (Sq) [nm]
CS immersed in 2.0M HCl only	1143	1269
CS immersed in 2.0M HCl and 300ppm PU (Sample)	96	129

Table 7. AFM data f	for C-S surface	with and without H	U environment.
	-		

#### 3.7. Mechanism of corrosion hindrance.

From the obtained results, the *PU* extract can be adsorbed on CS surface via physical and chemical adsorption.



Scheme 1. Corrosion inhibition mechanism.

"In physical adsorption, most of the extract molecules in HCl exist in protonated form (cation) which be in equilibrium with the conforming neutral form as:  $[PU] + H^+ \leftrightarrow [PUH. Cl^-$  ions are first adsorbed on positively charged CS surface and create an excess negative charge towards the solution side of the metal and favor more adsorption of cations, then protonated extract molecules adsorb via electrostatic interactions and form a protective layer [40,41]. These complexes adsorbed onto the CS by force (Van Der Waals) to form protecting cover to prevent CS from corrosion". This *PU* will present in the protonated form, so it can adsorb directly on the negative surface of CS in an acidic medium by electrostatic attraction, as shown in Scheme 1.

#### 5. Conclusions

The examined PU extract shows high inhibition efficiency for CS corrosion in a solution of 2 HCl, and the inhibition was clarified by the adsorption fundamental. The adsorption of PU extract over CS surface obeyed Langmuir adsorption isotherm. The adsorption parameters indicated that the adsorption of PU extract on the CS surface was spontaneous. PDP procedures indicate that PU extract exhibits cathodic and anodic inhibition effects. AFM results for CS surface with PU extract indicate low roughness, meaning that the inhibitive film is formed on the surface of the metal.  $C_{dl}$  diminishes while  $R_{ct}$  rises by increasing the doses of PU, which can be attributed to the adsorbed PU molecules. There is good agreement between chemical and electrochemical techniques.

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## **Conflicts of Interest**

The authors declare no conflict of interest.

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