

Adsorption and inhibitive impact of 5-(2-ethoxybenzylidene) 1,3-dimethylbarbituric acid on carbon steel corrosion in acid solution

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Abstract: The inhibiting impact of ecofriendly 5-(2-ethoxybenzylidene) 1,3-dimethylbarbituric acid (5-EBMB) in 1 M HCl on the corrosion of C-steel has been examined via weight loss (WL) method, potentiodynamic polarization (PP), electrochemical impedance spectroscopy (EIS), electrochemical frequency modulation (EFM) techniques. The attained outcomes exhibit that the investigated compound is excellent inhibitor and its inhibition efficiency (%IE) rises by rising concentration and temperature. The adsorption of the investigated derivative on the surface of C-steel follows Langmuir isotherm. The adsorption process of the investigated compound is spontaneous and considered as chemisorption type. PP curves revealed that the studied derivative is mixed-type inhibitor. Moreover, EIS results confirmed the adsorption of the investigated compound on C-steel surface via increasing on charge transfer resistance (R_{ct}) and lowering in capacitance of double layer (C_{dl}). The IE% of the investigated derivative reached to 79.9% at concentration 21×10^{-6} M according to WL method. All tested methods gave good agreement.

keywords: Carbon steel, Adsorption, inhibitive, corrosion

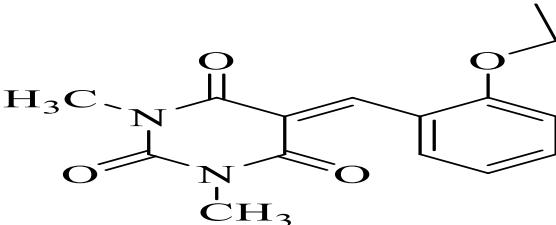
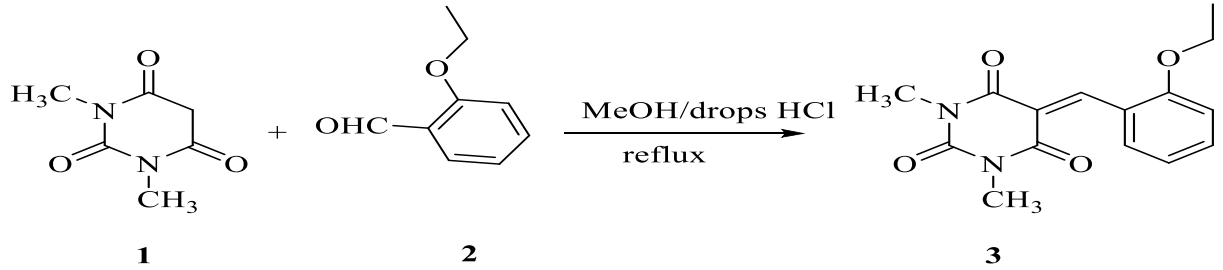
1. Introduction

Acidic media are generally applied for elimination of unwanted scale and corrosion in several industrial procedures. By monitoring metal dissolution attributable to acidic exposure, so inhibitors are commonly applied within this operation [1]. Organic inhibitors today do the inhibition of corrosion well than the inorganic inhibitors [2]. Organic compounds are kind of acidic inhibitors including hetero atoms for example oxygen, sulfur, and nitrogen. Amongst, organic inhibitors have several advantages for instance low cost, low poisonousness, high inhibition efficiency, and easy to organize [3-6]. Generally, heterocyclic organic compounds are applied to the corrosion inhibition on copper [7] aluminum [8-10], iron [11-16] and also other metals [17-18] within diverse corrosion media. A review of the literature on acid corrosion inhibitors reveal that they work by adsorbing to the metal's surface. This effect may be caused by (i) electrostatic attraction between the charged metal and the charged inhibitor molecules, (ii) dipole-type interaction between

uncharged electron pairs in the inhibitor and the metal, (iii) electron-interaction with the metal, or (iv) A combination of the aforementioned [19]. Pyrimidine is a six-membered heterocyclic aromatic compound with two nitrogen atoms at positions 1 and 3. The chemistry of pyrimidine derivatives is crucial in medicine, agrochemicals, and a variety of biological activities. Many well-known commercial medications contain pyrimidine derivatives, such as Uramustine, Piritrexim, Isetionate, Tegafur, Floxuridine, Fluorouracil, Cytarabine, and Methotrexate. Furthermore, the pyrimidine skeleton is found in a wide range of natural products, including nucleic acids, vitamins, enzymes, chlorophyll, hemoglobin, and hormones. Ansari et al [20] investigates the corrosion protection of mild steel by four PPDs in 1 M HCl solution and they found the percentage inhibition efficacy 88-97.1% at 400 mg L^{-1} . Numerous pyrimidine derivatives have been synthesized and studied their suitability for corrosion inhibition of variety of steel samples in acidic medium [21-27]. The efficacy

of the organic compounds including hetero atoms as corrosion inhibitors in acidic solutions for C-steels is well recognized [28-32]. Pyrimidines and their derivatives are important because they are available in nature, particularly in the nucleobases present in nucleic acids, and many of them have been discovered to be beneficial in chemotherapy [33]. Currently in use as anticancer, antifungal, and antibacterial medicines are pyrimidine-containing chemotherapeutics [34]. Furthermore, in HCl and H₂SO₄ solutions, several pyrimidine derivatives were found to be efficient corrosion inhibitors for steel [35].

Table 1. Chemical structures of tested inhibitor

Inhibitor Code	Molecular structures/Chemical name	Chemical Formula/Molecular Weight
MA-1020	 <p>5-(2-Ethoxybenzylidene)-1,3-dimethylbarbituric acid</p>	<p>C₁₅H₁₆N₂O₄ 288.3</p>
 <p>Scheme 1: Synthetic route of 5-(2-ethoxybenzylidene)-1,3-dimethylbarbituric acid</p>		

2.2. Inhibitor

5-(2-Ethoxybenzylidene)-1,3-dimethylbarbituric acid (5-EBMB) was prepared as outlined in **scheme 1**. The detailed information of 5-EBMB inhibitor is reported in the literature [36] with identical melting point of 165-166°C as in the Literature [37].

2.3. Electrochemical techniques

Electrochemical measurements are taken within traditional three electrodes glass cell include saturated calomel electrode (SCE) linked with fine “Luggin capillary, platinum counter electrode and working electrode is carbon steel with a square cut shape and surface area of 1.0×1.0 cm². PP curves are established

purpose of this work is to study the impact of 5-(2-ethoxybenzylidene)-1,3-dimethylbarbituric acid as ecofriendly inhibitor for C-steel in 1 M hydrochloric acid solution by applying WL, PP, EIS, EFM tests. The investigated 5-arylidene barbituric acid derivative is not reported as a corrosion inhibitor for steel in the literatures.

2. Materials and methods

2.1. Materials

Chemical composition of C-steel samples in weight percentage are carbon (0.200%); manganese (0.350%); phosphor (0.024%); chromium; sulfur (0.003%); and balance

through altering the electrode potential automatically from -500 to +500 mV vs. OCP with a sweep rate of 1 mVs⁻¹. Stern-Geary method [40] applied the definition of corrosion current is achieved via deducing on cathodic and anodic Tafel lines to a point which provides log *i*_{corr} and the resulting E_{corr} for inhibitor free acid and to any concentration of inhibitor”. Thereafter *i*_{corr} can be applied to examine of θ and IE % as subsequent:

$$IE\% = \theta \times 100 = \left[1 - \frac{i_{corr}}{i_{corr(inh)}} \right] \times 100 \quad (2)$$

Where, “*i*_{corr(free)} and *i*_{corr(inh)} are the corrosion current densities at the absence and existence of inhibitor”, separately.

EIS are applied within range of frequency from 100 kHz to 0.01 mHz and 10 mV amplitude peak-to-peak at OCP. The θ and the IE% achieved from the impedance calculation were assessed through the next equation:

$$IE\% = \theta \times 100 = \left[1 - \frac{R_{ct}^{\circ}}{R_{ct}} \right] \times 100 \quad (3)$$

Where, " R_{ct}° and R_{ct} are the resistance of charge transfer at the absence and existence of inhibitor", separately.

EFM tests were accomplished via dual frequencies "2 and 5 Hz with base frequency 0.1 Hz, consequently the wave shape repeats subsequently at 1 s. The large peaks located in the intermodulation spectra were utilized to assess the corrosion current density (i_{corr}), the Tafel slopes (β_a and β_c) and CF-2 & CF-3" [41, 42], the %IE and θ were assessed from Equation (2).

All electrochemical experiments are ready solution at $25 \pm 1^{\circ}\text{C}$. The potential of electrode can be permitted until become stable 30 min prior to start the measurements. All electrochemical experiments were done at $25 \pm 1^{\circ}\text{C}$ and accomplished via Gamry (PCI4/750G) Potentiostat/Galvanostat/ZRA. This includes Gamry Framework for controlling and Echem Analyst5.58 software for data analysis and plotting.

3. Results and Discussion

3.1. WL method

The WL-time diagrams for the corrosion of C-steel in 1 M hydrochloric solution before and after addition of diverse concentrations of inhibitor is displayed within Fig(1). Fig (1) demonstrates that the values of WL for C-steel with 1M hydrochloric acid solution lies higher than in inhibitor and the WL decreases as inhibitor dose rises; It is meaning the strengthens of corrosion inhibition by increasing the inhibitor concentration as listed in Table (2). This explains the adsorption of inhibitor molecules on the C-steel surface, i.e., the C-steel surface is shielded from the aqueous media through creation of protecting film on this surface [43, 44].

3.2. PP studies

Figure 2 illustrates the Tafel polarization diagrams for C-steel in 1 M hydrochloric acid

in the absence and existence of inhibitor dose at 25°C , separately. From Fig.2, it is obvious that anodic metal dissolution and cathodic H_2 reduction reactions were controlled when examined inhibitor was added to 1 M HCl solution also this inhibition was more obvious through rising the dose of the inhibitor. Table 3 illustrates that i_{corr} declines via addition of the inhibitor and through raising its doses. Furthermore, E_{corr} does not change clearly, and this exhibits that the examined derivative is considered as mixed-type inhibitor [45, 46]. Moreover, Tafel slopes [β_a , β_c] are almost constant indicating that the two reactions (i.e., anodic metal dissolution and cathodic hydrogen reduction) were slightly affected without altering mechanism of dissolution [47, 48].

Table 2. Variation of %IE with altered doses of investigated inhibitor at 25°C from WL measurements at 120 min dipping in 1.0 M HCl.

Compound	Conc. (M)	CR ($\text{mg cm}^{-2} \text{min}^{-1}$)	%IE
Blank	---	0.028	---
5-EBMB	1×10^{-6}	0.022	47.1
	5×10^{-6}	0.017	55.8
	9×10^{-6}	0.015	58.1
	13×10^{-6}	0.013	62.7
	17×10^{-6}	0.011	68.6
	21×10^{-6}	0.009	74.7

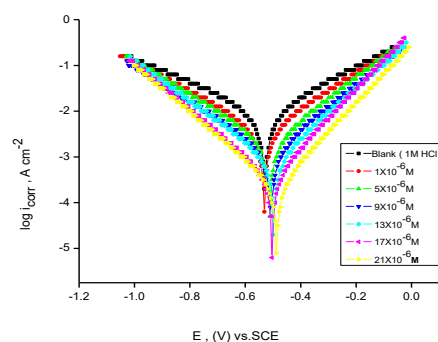


Fig. 1 Time-WL bends for C-steel in 1M HCl in the absence and presence of diverse doses of 5-EBMB at 25°C

3.3. EIS studies

The impact of the doses of inhibitor on the impedance of C-steel in 1M HCl at 25°C is produced in Fig. 3 [a, b]. Curves show identical kind of Nyquist bends for C-steel with existence of diverse doses of 5-EBMB. Presence of single semi-circle displayed the single charge transfer procedure through

dissolution which is unaltered with the existence of inhibitor compound. Deviations from ideal circular form are frequently signalize to the frequency dispersal of

impedance interfacial which occurs because of impurities, surface coarseness, grain limits, dislocations, forming of porous layers and

Table 3. Corrosion parameters of C-steel electrode in 1M HCl solution containing altered doses of 5-EBMB at 25°C from PP technique

Conc.,M	$-E_{corr}$ (mV,vs.SCE)	i_{corr} (mA cm ⁻²)	β_c mV dec ⁻¹	β_a mV dec ⁻¹	θ	%IE	CRmmy ⁻¹
1 M HCl	587	422	42	22	---	---	220.6
1x10 ⁻⁶	585	369	37	36	0.378	37.8	186.5
5x10 ⁻⁶	584	263	61	42	0.476	47.6	120.1
9x10 ⁻⁶	573	206	71	51	0.579	57.9	94.3
13x10 ⁻⁶	590	160	80	58	0.685	68.5	73.3
17x10 ⁻⁶	583	123	64	46	0.757	75.7	56.2
21x10 ⁻⁶	572	96.8	122	82	0.825	82.5	44.2

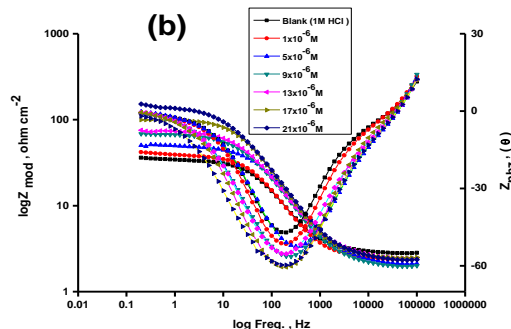
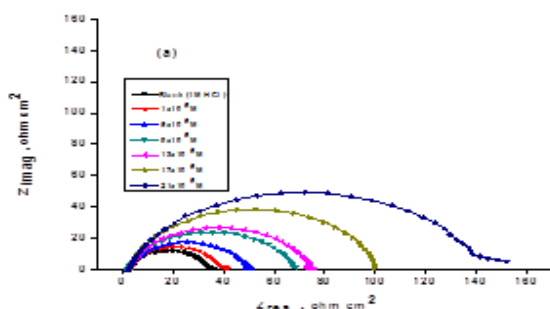


Fig. 2 PP diagrams for dissolution of C-steel in 1M HCl in the attendance and absence of altered doses of 5-EBMB at 25 °C. adsorption of derivatives, also homogenized on the surface of electrode [49,50]. Observation of these data detected from all impedance graphs contains of large capacitive circle by only time constant of capacitive with Bode–phase graphs (Fig.3b). The electrical equivalent circuit is displayed in Fig. 4 and it applied for examine achieve impedance data. This circuit involves R_{ct} , C_{dl} also the solution resistance (R_s). Fit excellent through this model can be gained through experimental data. EIS outcomes in Table 4 distinguished that C_{dl} values declines as well as R_{ct} values rises by rising doses of inhibitor. It is because of the increasing in the thickness of the adsorbed layer and to the exchange of the adsorbed water molecules with the inhibitor molecules on the surface of metal, declining the metal dissolution reaction [51, 52]. The diminishing in C_{dl} can be caused by a drop in the local dielectric constant and/or a rise in the thickness of the double layer electrical suggested that inhibitor molecule function through adsorption at the metal and solution interface [53]. The %IE gained from EIS studies are close to those inferred of PP studies.

Fig. 3 Nyquist (a) and Bode (b) plots for C-steel in 1M HCl at altered doses of 5-ABA at 25°C.

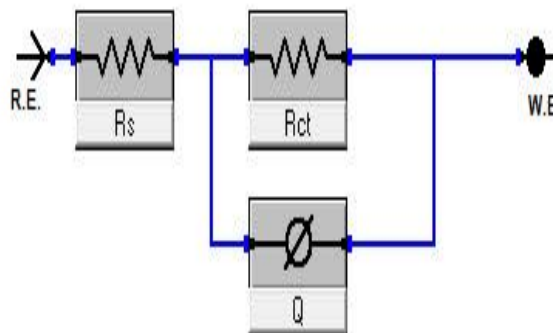


Fig. 4: Electrical equivalent circuit model utilized to fit the results of impedance

Table 4: EIS data of C-steel in 1M HCl and in existence of altered doses of 5-EBMB at 25 °C

Conc., M	C_{dl} (μ F cm ⁻²)	R_{ct} (ohm cm ²)	θ	%IE
1M HCl	117.9	31.7	---	---
1x10 ⁻⁶	102.5	37.5	0.147	14.7
5x10 ⁻⁵	92.3	47.8	0.339	33.9
9x10 ⁻⁵	86.1	66.1	0.521	52.1
13x10 ⁻⁶	79.1	72.5	0.563	56.3
17x10 ⁻⁶	77.4	97.7	0.676	67.6
21x10 ⁻⁶	69.8	139.7	0.773	77.3

3.4. EFM studies

EFM spectra intermodulation for C-steel in 1 M hydrochloric acid solution before and after adding $21 \times 10^{-6} \text{ M}$ of 5-EBMB is displayed in **Fig. 5**. The bigger peaks were applied to examine i_{corr} , β_c , β_a , CF-2 and CF-3. Those electrochemical factors are concurrently specified then recorded in **Table 5**. It can be viewed from this **Table 5**, the values of i_{corr} diminish with existence of various doses of 5-EBMB than with existence only of 1 M HCl in situation of C-steel. The obtained Causality factors for the examined data are in excellent quality with their theoretical (2 &3) values.

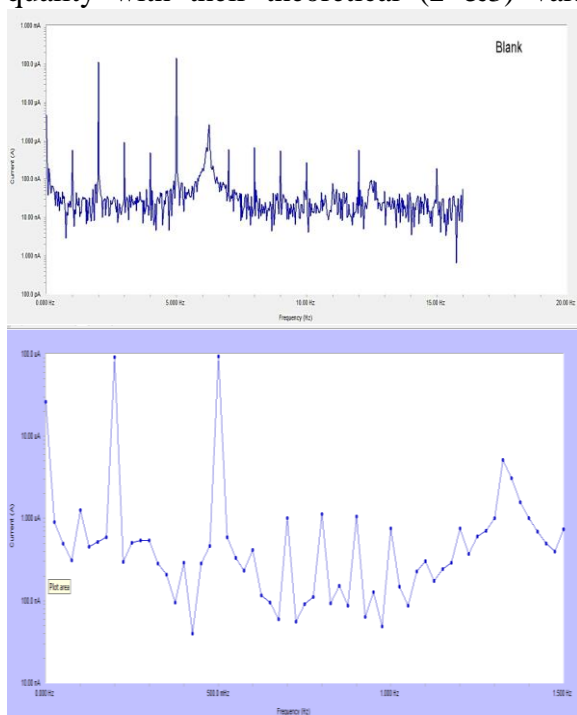


Fig. 5 EFM spectra for C-steel in 1M HCl with and without $21 \times 10^{-6} \text{ M}$ of 5-ABA at 25°C

3.5 Effectiveness of temperature

Temperature impact on the rate of corrosion of C-steel in 1 M HCl including diverse concentration of the investigated inhibitor can be examined via WL method at temperature

Table 5: EFM parameters for C-steel 1M HCl solution and existence of altered doses of 5-EBMB at 25°C

Conc.,(M)	$i_{\text{corr}}(\mu\text{A cm}^{-2})$	$\beta_1(\text{mVdec}^{-1})$	$\beta_2(\text{mVdec}^{-1})$	CF-2	CF-3	CR mmy^{-1}	% IE
1M HCl	808.5	113	164	2.09	1.75	370.2	---
1×10^{-6}	599.9	99	137	2.04	3.23	274.1	25.8
5×10^{-6}	453.9	101	126	1.95	3.09	207.4	43.9
9×10^{-6}	374.5	112	118	2.05	3.08	172	53.7
13×10^{-6}	272.7	97	101	1.73	2.61	124.6	66.3
17×10^{-6}	233.1	92	118	1.97	2.57	106.5	71.2
21×10^{-6}	152.6	105	111	1.49	1.83	69.7	81.1

ranges of 25 to 55°C (**Table 6**). The Outcomes discovered that, by raising the temperature the rate of corrosion rises and decline with dose of the 5-EBMB rise for the investigated inhibitor.

The activation energy (E_a^*) can be examined by applying Arrhenius equation:

$$k = A e^{\frac{-E_a^*}{RT}} \quad (4)$$

where, “A is Arrhenius constant and k is rate of corrosion. Straight lines are displayed in **Fig. 6**.and their linear regression (R^2) is nearer to 1 and E_a^* can be obtained from the slope. Table 6 displayed that the value of E_a^* for uninhibited solution is lower than inhibited solution, supposing that the dissolution of C-steel is slow within existence of inhibitor [54]. This is recognized from Eq. 10 the higher values of E_a^* cause lower corrosion rate owing to construction of protecting film on the C-steel surface acting as an energy barrier of the C-steel corrosion [55-57]. Entropy and enthalpy of activation (ΔS^* , ΔH^*)” of the corrosion procedure were reckoned from the transition state theory:

$$k = \left[\frac{RT}{Nh} \right] e^{\frac{\Delta S^*}{R}} e^{\frac{-\Delta H^*}{RT}} \quad (5)$$

where, “N Avogadro's number and h Planck's constant”. The graphs of $\log k/T$ versus $1/T$ of C-steel with 1 M hydrochloric acid solution at diverse doses from examined compound, provides straight lines as displayed in **Fig.7** for inhibitor. The thermodynamic parameters are list in **Table 6** shows that ΔH^* values are positive signalize that the steel dissolution process is endothermic process. High and negative values of ΔS^* assume that activated complex found in an association form more than dissociation form.

Table 6: Data of WL measurements for C-steel in 1M HCl solution with and without altered doses of 5-EBMB at 25 – 55°C

Inhibitor	Conc. (M)	Temp. (°C)	CR (mg cm ⁻² min ⁻¹)	θ	%IE
5-EBMB	Blank (1 M HCl)	25	0.028	----	----
		35	0.033	----	----
		45	0.039	----	----
		55	0.045	----	----
	1x10 ⁻⁶	25	0.022	0.249	24.9
		35	0.026	0.196	19.6
		45	0.033	0.149	14.9
		55	0.038	0.137	13.7
	5x10 ⁻⁶	25	0.017	0.394	39.4
		35	0.022	0.314	31.4
		45	0.028	0.258	25.8
		55	0.033	0.242	24.2
	9x10 ⁻⁶	25	0.015	0.471	47.1
		35	0.019	0.405	40.5
		45	0.025	0.339	33.9
		55	0.030	0.315	31.5
	13x10 ⁻⁶	25	0.013	0.558	55.8
		35	0.017	0.475	47.5
		45	0.022	0.416	41.6
		55	0.026	0.403	40.3
	17x10 ⁻⁶	25	0.011	0.627	62.7
		35	0.014	0.554	55.4
		45	0.019	0.495	49.5
		55	0.023	0.479	47.9
	21x10 ⁻⁶	25	0.009	0.683	68.3
		35	0.012	0.627	62.7
		45	0.017	0.563	56.3
		55	0.020	0.555	55.5

Table 7: Activation parameters for dissolution of C-steel in the absence and existence of altered doses of 5-EBMB in 1M HCl

Inh.	Activation parameters				Regression Coefficient(R ²)
	Conc. M	E _a *,kJmol ⁻¹	ΔH*,kJ mol ⁻¹	ΔS*, -J mol ⁻¹ K ⁻¹	
5-EBMB	Blank 1M HCl	12.2	9.6	108.6	0.9941
	1x10 ⁻⁶	15.4	12.8	99.8	0.9921
	5x10 ⁻⁶	17.6	15.1	94.1	0.9917
	9x10 ⁻⁶	19.3	16.8	89.7	0.9868
	13x10 ⁻⁶	19.9	16.9	90.3	0.9907
	17x10 ⁻⁶	21.3	18.8	85.7	0.9845
	21x10 ⁻⁶	21.7	19.6	84.2	0.9718

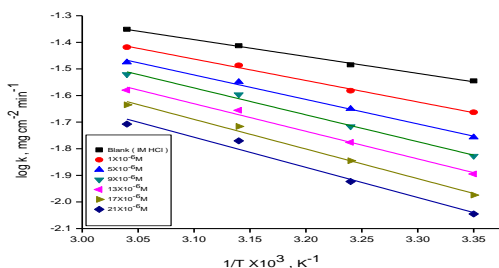


Fig. 6: Log k – 1/T curves for C-steel dissolution in 1.0 M HCl in the absence and existence of altered doses of 5-EBMB.

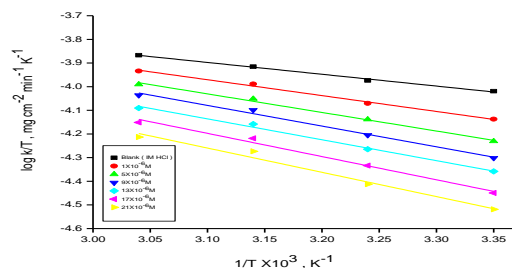
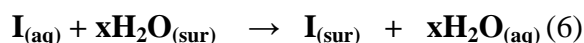


Fig. 7: log k/T – 1/T curves for C-steel dissolution in 1M HCl and existence of altered doses the investigated 5-EBMB

6. Adsorption Isotherm

Organic compounds are inhibited the metal corrosion through adsorption on surface of metal. The adsorption procedure is considered as single replacement process of adsorbed water molecules (x) by a single inhibitor molecule [58, 59].



As well, the adsorption affords data about interaction between the adsorbed molecules and the surface of metal. The values of θ for diverse doses of the analyzed inhibitor at various temperatures have been applied to describe the most suitable adsorption isotherm to define adsorption procedure. Results of the studied derivative are suitable with Langmuir adsorption isotherm. **Fig 8** displays the plotting of C/θ versus C at 25°C to examine inhibitor, separately. Those schemes provided straight lines with unit slope signaled that adsorption

of 5-EBMB on C-steel surface confirmed Langmuir equation [60].

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \quad (7)$$

where, “ C is the inhibitor concentration and K_{ads} is adsorption equilibrium constant” associated to the free energy of adsorption ΔG_{ads} as appear [61]:

$$K_{ads} = \frac{1}{55.5} e^{\frac{-\Delta G_{ads}^{\circ}}{RT}} \quad (8)$$

where, “ T is the absolute temperature R is the universal gas constant and 55.5 is the concentration of water on the metal surface in M ”. The data of K_{ads} and ΔG_{ads}° for 5-EBMB are listed within **Table 7**. The increase in the negative value of ΔG_{ads}° indicates that the 5-ABA was strongly adsorbed onto the C-steel surface in a stable state and that the adsorption process was spontaneous. Furthermore, the values of ΔG_{ads}° are -32.5 and -33.0 kJ mol^{-1} indicate that the adsorption of 5-EBMB on C-steel is mixed type i.e., physisorption and chemisorption, but mainly physisorption because the E_a^* values increases in presence of inhibitor than in its absence and % inhibition decreases by raising temperature. [62].

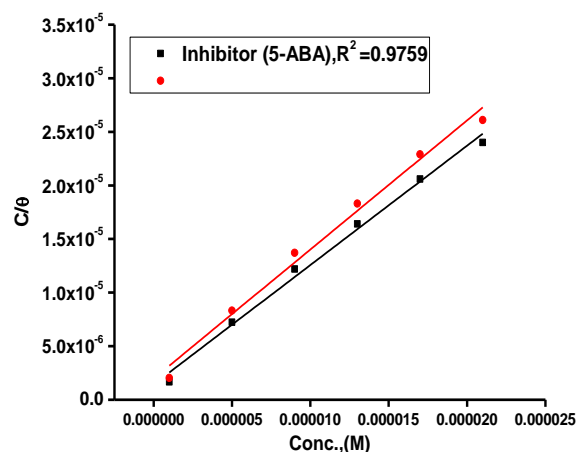


Fig. 8: Langmuir isotherm plots for C-steel in 1 M HCl containing various doses of 5-EBMB at 25 °C.

Table 8: Equilibrium constant and adsorption free energy of 5-EBMB adsorbed on C-steel surface at 25 °C

Langmuir isotherm				
Inhibitor	$K \times 10^{-5}, M^{-1}$	$-\Delta G_{ads}^{\circ}, \text{kJ mol}^{-1}$	Slope	R^2
5-EBMB	1.82	40.0	1.28 5	0.97 59

4. Conclusions

5-Arylidene barbituric acid derivative is considered as good inhibitor for the corrosion of C-steel in 1 M HCl as approved by experimental and theoretical studies. The adsorption of the 5-EBMB on C-steel surface is followed Langmuir isotherm and deemed as mixed type but mainly physisorption. PP results revealed that the investigated 5-EBMB hindered anodic and cathodic reactions jointly (i.e., mixed type inhibitor). The values of C_{dl} decline and R_{ct} rise compared to blank solution when the inhibitor is existed, confirming the adsorption of inhibitor molecule on the surface of C-steel.

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