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EXPERIMENTAL STUDY ON THE INHIBITORY EFFECT OF EXPIRED CHLORPROMAZINE ON CORROSION OF MILD STEEL IN ACIDIC MEDIUM

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ABSTRACT

Chlorpromazine was investigated for its corrosion inhibition potential on mild steel in 0.5 M H_2SO_4. Weight loss measurement and electrochemical impedance spectroscopy (EIS) methods were used. The concentrations of chlorpromazine ranges from (0.2 g to 1.0 g) and temperatures (298 – 333 K) at 1 hour on corrosion in acid medium and corrosion inhibition was assessed. The results showed that chlorpromazine decreased the corrosion rate of mild steel in the acid media. The rate generally decreased with increasing the concentration of chlorpromazine in acidic medium for mild steel at different temperature (298 K-333 K) at 1 hour. The maximum inhibition efficiency was 93.66 % at 298 K 0.5 M H_2SO_4 for 1 h on mild steel. As the temperature increases the inhibition efficiency decreases (333 K), suggesting physical adsorption of the chlorpromazine constituents on the metal surfaces. Electrochemical impedance spectroscopy was used as a method to investigated chlorpromazine in acidic medium. As the concentration of the expired chlorpromazine increases, the radius and size of semicircles increases. This demonstrates that the characteristics of the coating film on the surface of the mild steel increased thereby reduced the corrosion rate.

Keywords: Corrosion inhibition, Chlorpromazine, Mild steel, H_2SO_4, Adsorption, Thermodynamics

INTRODUCTION

Corrosion has damage a lot of metals in many industries such as pipelines, rail, installation of plants, transportation, and infrastructure. In these fields, new metallic materials are being continuously developed, with properties that more closely match the proposed objectives and are obviously more affordable. This justifies further research on corrosion processes order to identify new protection methods against corrosion. Corrosion is an electrochemical process that returns metals to their natural states through deterioration (Eddy *et al*.,2020). Corrosion is oxidation and reduction. The reaction of corrosion at anode and cathode.

$$Fe_{(S)} \longrightarrow Fe^{2+}_{(Aq)} + 2^{e-} \quad (1)$$

$$_{1/2}O_2 + 2H_2O + 4e \longrightarrow 2OH \quad (2)$$

$$Fe^{2+}(OH)_2 \longrightarrow Fe(OH)_2 \quad (3)$$

$$4Fe(OH)_2 + 2H_2O + O_2 \longrightarrow 4Fe(OH)3 \quad (4)$$

$$2Fe(OH)_3 \longrightarrow 2H_2O + Fe_2O_3.H_2O \quad (5)$$

Corrosion cannot be eliminated completely, but different methods are used to slow the rate of corrosion (Fayemi *et al*., 2022). Consequently, industrial facilities exposed to aggressive medium like acid, base and salt are often protected from corrosion by adopting several method including painting, oiling, etc. The use of inhibitors has been found to be one of the best method available for the protection of metals against corrosion (Ebenso *et al*., 2020). Corrosion inhibitors are chemical substances that, when added in a minute amount to an aggressive environment, mitigate the metal degradation process. They are compounds that tend to retard the rate of corrosion of metals through adsorption on the surface of the metal either by charge transfer from charged inhibitor molecule to charged metal surface (physical adsorption) or by electron transfer from the inhibitor's molecule to the vacant d-orbital of the metal (Varma *et al*., 2018). (Ebenso *et al*., 2018).

Chlorpromazine has given hope of meeting environmental requirements for corrosion inhibitors. Chlorpromazine has chemical formula $C_{17}H_{19}ClN_2S$ and structural formula of chlorpromazine.

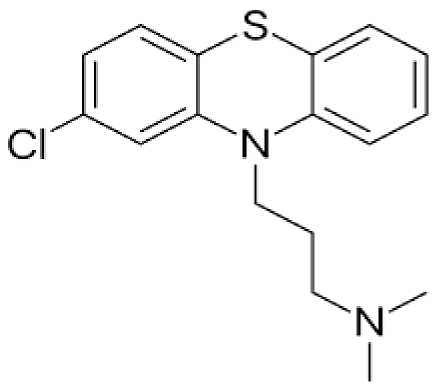

Figure 1: Chemical Structure of Chlorpromazine
Chemical formula: $C_{17}H_{19}ClN_2S$
Molar mass: 318.86 g/mol.

Reaction between chlorpromazine and mild steel in acid medium

$$Fe + H_2SO_4 \longrightarrow FeSO_4 + H_2 \quad (6)$$

$$C_{17}H_{19}ClN_2S + Fe^{2+} \longrightarrow [Fe\,(C_{17}H_{19}ClN_2S)_2]^{2+} + H_2^{+} \quad (7)$$

$$Fe + 2C_{17}H_{19}ClN_2S + 2H_2SO_4 \longrightarrow [Fe\,(C_{17}H_{19}ClN_2S)_2]^{2+} + FeSO_4 + 2H_2 \quad (8)$$

MATERIALS AND METHODS

Experimental procedure

Materials used for this study were mild steel, mild steel sheet was mechanically press-cut to form coupons, each of dimensions coupon were 5 cm x 2 cm x 1 cm for weight loss studies and 3 cm x 2 cm x 1 cm for electrochemical studies. The coupons was wet polished with different grades of abrasive paper (400-1200), washed with distilled water, rinsed with absolute ethanol, cleaned in acetone and allowed to dry in the air before being preserved in a desiccator prior to corrosion testing and monitoring.

All the reagents that was used for this research were prepared from the stock solution procured from Chemistry Store in the Department of Chemistry, Nigerian Defence Academy (NDA).

Sampling

Expired chlorpromazine drugs were collected randomly from different sources such as 44 Reference hospital, 461 Aeromedical center NAF base, Kaduna, Pharmaceutical stores. The samples were identified at National Agency for Food and Drugs Administration Control (NAFDAC) Kaduna and they were assigned a batch number for proper identification.

Expired chlorpromazine drug was crushed and grinded with ceramic mortar and pestle into a fine powder form, and they were preserved in a plastics container and labelled accordingly as sample pretreated. While mild steel was pretreated by polishing with abrasive paper, and cleansed with tissue paper and clean with cloth. Then wash with tap water, wash with distilled water and finally wash with acetone in order to degrease them, they were air dried and keep in desiccator.

Weight loss Method

Weighed mild steel was dipped into 250 mL containers that separately contains of 0.5 M H_2SO_4 for 1 hour without corrosion inhibitor been added to the 250 mL of 0.5 M H_2SO_4 and take the record accordingly at different temperature i.e. 298, 303, 313, 323 and 333 K this is known as a blank record. Weighed mild steel coupons was completely immerse in 250 cm^3 of the test solutions separately (0.5 M H_2SO_4) in an open beaker placed in a water bath maintained at different temperature i.e. 303, 313, 323 and 333 K respectively.

After 1 hour, each corrosion products was remove and washing each coupon (withdrawn from the test solutions) with distilled water. The washed coupons was rinsed in acetone and dried in air before re-weighing. From the initial and final weights of the specimens, the loss of weights was calculated, ΔW, as follows (Owen *et al.*, 2019).

$$\Delta W = \frac{M_1 - M_2}{A} \quad (9)$$

Where M_1 is the mass of the specimen before corrosion, M_2 the mass of the specimen after corrosion, and A the expose area of the specimen. The corrosion rate (in mmy^{-1}) was computed from the following equation:

$$CR = 87.6 \times W/_{DAt} \quad (10)$$

Where W is the weight loss in mg, D is the density of the specimen, A is the surface area of specimen (cm^2) and t is the time of exposure of the sample in hours.

The efficiency of the inhibitor was computed using the following equation Inhibition.

$$\%IE = \frac{CR\,blank - CR\,inhi}{CR\,blank} \text{ X } 100 \quad (11)$$

Where CR blank is the corrosion rate in blank and CR inhi is the corrosion rate with inhibitors.

Electrochemical measurements procedure

The electrochemical impedance spectroscopy was done at Defence Industries Corporation of Nigeria (DICON) Kaduna. The Mild steel sample was cut to 3 cm^2 shape for the electrochemical experiments. The coupons was polished with fine micro size emery paper, washed with distilled water, rinsed with absolute ethanol, cleaned in acetone, dried in the air and keep in a desiccator. One face of the specimens was connected to a copper wire to be use as working electrode. All electrochemical experiments was perform in a three-electrode cell, consisting of working, reference and the counter electrode. Here saturated calomel electrode and platinum electrode was used as reference and counter electrode respectively. The three electrodes was connected to the Electrochemical Workstation, CH Instruments model CH660C. All tests was perform in 0.5 M H_2SO_4 media at room temperature (298 ± 1 K) in presence and absence of inhibitor (blank). The percentage of inhibitions from impedance measurements was calculated using charge transfer resistance values by the following expression

$$n_{EIS}\% = \frac{R_{ct}}{R_{ct}} - \frac{R'_{ct}}{R_{ct}} \times 100 \quad (12)$$

Where R_{ct} and R'_{ct} are the charge transfer resistances of working electrode with and without inhibitor respectively.

Prior to polarization, the system was left undisturbed for an hour, which should be sufficient to attain stable open circuit potential (OCP).

RESULTS AND DISCUSSION

Weight Loss Measurement

The weight loss of mild steel in the presence and absence of chlorpromazine at different temperatures for 1 hour of immersions was determined by weight loss measurements. From the Table 1, it was evident that there was metal dissolutions in the presence and absence of the chlorpromazine inhibitor. It is important to note that the weight loss in the absence of the chlorpromazine was higher than that in the presence of the chlorpromazine. In general, weight loss was found to decrease steadily with increase in the concentration of the chlorpromazine. The rate of weight loss on mild steel in the absence and presence of the chlorpromazine is shown in Table1. The weight loss is higher in the blank solution than in the solution containing the chlorpromazine. The maximum weight loss rate recorded in the blank was 624.3344 mg on the 0.5 M H_2SO_4 acid at 1 hour 333 K.

Table 1: Corrosion Rate, Surface Coverage and Inhibition Efficiency of chlorpromazine on mild steel in 0.5 M conc. H_2SO_4 for 1 hour at different temperature

Temp.(*K*)	System (g)	CR (mm/y)	%IE	(*θ*)
298 K	Blank	138.7768	_	_
	0.2	62.9337	54.65	0.5465
	0.4	43.3390	68.77	0.6877
	0.6	34.5789	75.08	0.7508
	0.8	29.9684	78.41	0.7841
	1.0	13.8316	93.66	0.9366
303 K	Blank	263.4916	_	_
	0.2	126.5589	51.97	0.5197
	0.4	84.3726	67.98	0.6798
	0.6	60.6284	76.99	0.7699
	0.8	42.6424	83.81	0.8381
	1.0	29.2768	88.89	0.8889
313 K	Blank	368.8421	_	_
	0.2	223.3800	39.44	0.3944
	0.4	169.2063	54.13	0.5413
	0.6	121.4874	67.06	0.6706
	0.8	100.9705	72.63	0.7263
	1.0	58.3232	84.19	0.8419
323 K	Blank	509.5323	_	_
	0.2	273.4042	46.34	0.4634
	0.4	188.8011	62.95	0.6295
	0.6	146.1537	71.32	0.7132
	0.8	127.0200	75.07	0.7507
	1.0	86.2168	83.08	0.8308
333 K	Blank	624.3344	_	_
	0.2	331.7274	46.87	0.4687
	0.4	267.8716	57.09	0.5709
	0.6	204.4768	67.25	0.6725
	0.8	138.3158	77.85	0.7785
	1.0	114.5716	81.65	0.8165

Gravimetric analysis

Weight loss increases as the temperature increase, but as the concentration of the inhibitor increase, the corrosion rate decrease. Therefore, the corrosion rate of the metal is decreased due to the presence of chlorpromazine, and the inhibition efficiency increases with increase in the concentration of chlorpromazine. The chlorpromazine acted as an adsorption inhibitor in that the extent of coverage increases with concentration (Owen *et al*., 2019). Corrosion rate was also found to increase with increase in temperature which agrees with the mechanism of chemisorption, since the extent of adsorption increase with temperature, contrary to physiosorption (in which the extent of adsorption/inhibition efficiency decreases with increase in temperature (Awe, 2017) (Nkuna *et al*., 2020).

Kinetics study

The Arrhenius equation was used to investigate the effect of temperature on the inhibition of the corrosion of mild steel by chlorpromazine. The mathematical expression of the Arrhenius equation can be written according to equation 5 (Awe *et al*., 2019)

$$CR = A \exp \frac{-Ea}{RT} \qquad (13)$$

CR denote corrosion rate of mild steel, A is the Arrhenius or pre-exponential factor, R is the gas constant and T is the temperature. Widely applied form of the Arrhenius equation engages the plotting of ln(CR) versus 1/T to obtained slope equal to Ea/R and intercept equal to lnA. The Arrhenius plots for the corrosion of mild steel in the presence of chlorpromazine are shown in Fig. 1 while Arrhenius parameters obtained from the plots are recorded in Table 2

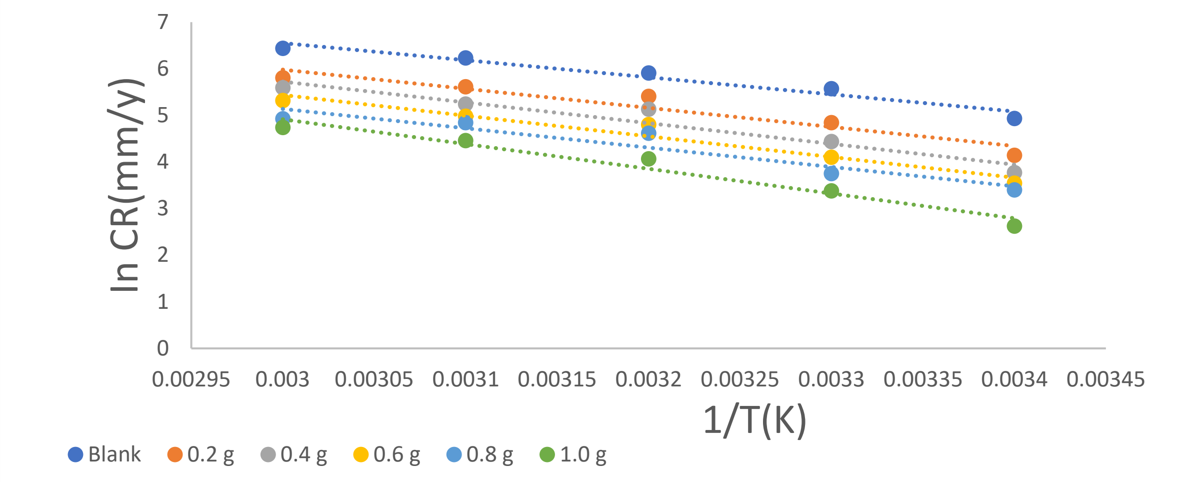

Figure 2: Arrhenius plots for the corrosion of mild steel in 0.5 M H_2SO_4 for 1 hour in the presence and absence of chlorpromazine at different temperatures

Table 2: Arrhenius parameters for the adsorption of chlorpromazine on mild steel surface 0.5 M H_2SO_4 for 1 hour H_2SO_4

System	Slope	In A	Ea (k/jmol)	A	R
Blank	-3667.1	17.552	30.49	41950461.84	0.9558
0.2 g	-4094.7	18.264	34.04	85497697.18	0.922
0.4 g	-4448.3	19.068	36.98	191041364.1	0.9338
0.6 g	-4434.5	18.741	36.87	137756749.1	0.956
0.8 g	-4150.1	17.589	34.50	43531701.47	0.9063
1.0 g	-5308.5	20.841	44.13	1124945020	0.9622

Thermodynamic study

Thermodynamic parameters is very important to determine the nature of adsorption in corrosion study, understanding the feasibility of the adsorption and the heat content of the adsorption process. Entropy and enthalpy data for the inhibition of the corrosion of mild steel in 0.5 M H_2SO_4 were also obtained through the slope and intercept of the transition state plots respectively. The transition state equation relates the corrosion rate to standard entropy and enthalpy of adsorption as follows (Omogbehin *et al*, 2024).

$$\ln = \left(\frac{CR}{T}\right) = In\ \frac{R}{Nh} + \frac{\Delta S0ads}{R} - \frac{\Delta H0ads}{RT} \quad (14)$$

A high degree of linearity was observed for plots of ln (CR/T) versus 1/T . Transition state plots for various concentrations of the inhibitor (and that of the blank) are presented in Fig. 3 while calculated values of $\Delta H^{O}ads$ and $\Delta S^{O}ads$ are recorded in Table 3. Standard enthalpies of adsorption of the inhibitors were positive while standard values of entropy change were negative. Therefore, the adsorption of the inhibitor is endothermic and is facilitated in the direction of increasing order.

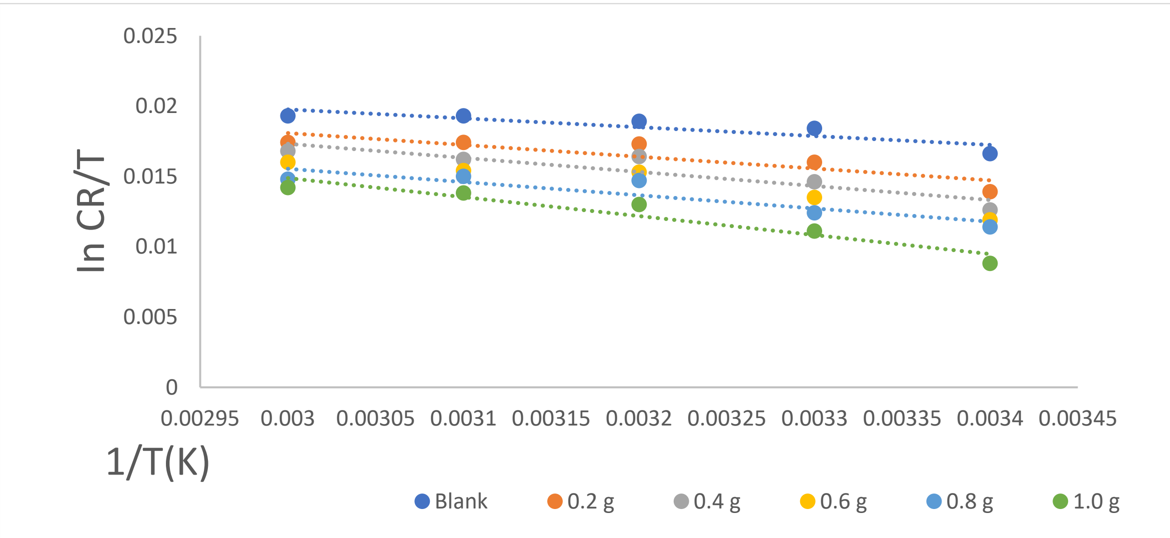

Figure 3: Transition state plots for the adsorption of chlorpromazine on mild steel surface 0.5 M H_2SO_4 for 1 hour

Table 3: Thermodynamic parameters for the adsorption of chlorpromazine on mild steel surface 0.5 M H_2SO_4 for 1 hour

System	slope	intercept	ΔH^o_{ads} (J/mol)	ΔS^o_{ads} (J/mol)	R^2
Blank	-6.3	0.0387	52.38	-197.22	0.7844
0.2 g	-8.4	0.0433	69.84	-197.18	0.7653
0.4 g	-10	0.0473	83.14	-197.15	0.83
0.6 g	-10.1	0.0467	83.97	-197.15	0.8926
0.8 g	-9.4	0.0437	78.15	-197.18	0.8127
1.0 g	-13.5	0.0554	112.24	-197.08	0.9127

Adsorption isotherm is very important during corrosion studies, they give descriptive mechanism on how inhibitor is adsorbed on the metal surface (Mobi *et at*., 2023).

Adsorption of organic molecules occurs when the interaction energy between the metal surface and organic molecule is higher than that of metal surface and water molecule. It is evident in Figs. 4 and 5 that Freundlich and Temkin adsorption isotherm gave best fitted isotherms for the adsorption of chlorpromazine on the surface of mild steel (with R^2 values above 0.9).

The Temkin adsorption model can be written as

Temkin: $\theta = \text{In } C + \text{In } K$ (15)

Where 'a' is the interaction parameter, θ is the surface coverage of the inhibitor, kads is the Temkin adsorption equilibrium constant and C is the concentration of inhibitor in (g).

Simplification of equation 7 yielded equation 8,

$$\theta = \frac{-1}{2a\ inkads} + \frac{-1}{(2a)InC} \quad (16)$$

On the other hand, the Freundlich adsorption equation can be written according to equation 9, which is transformed to equation 10

$$\theta = \text{kadsC}\,\frac{1}{n} \quad (17)$$

$$ln\theta = lnkads + \frac{1}{n\ InC} \quad (18)$$

Based on equations 8 and 10, Temkin and Freundlich adsorption isotherms were plotted and are shown in Figs. 4 and 5 respectively. Slope, intercept, Temkin and Freundlich parameters are presented in Table 4.

The results indicated that there is a repulsive behaviour of the inhibitor's molecules since the interaction parameters were negative. Also from the Fruendlich parameter (n), it is observed that the factor decreases with increase in temperature, indicating that the number of inhibitor's molecules that is adsorbed on the surface of the metals decreases with increasing temperature.

The Fruendlich and Temkin adsorption parameters were also used to calculate the standard free energy of adsorption of the inhibitor on the surface of the metal. These constants are related to the free energy of adsorption according to equation 11,

$$\Delta G_O ads - (55.5kads) \quad (19)$$

The free energy changes as recorded in Table 4 indicate that the adsorption of the inhibitor is spontaneous (since values of $\Delta Gads*$ are negative) and followed the mechanism of physical adsorption (because the $\Delta Gads*$ are less than the threshold value of -40 kJ/mol).

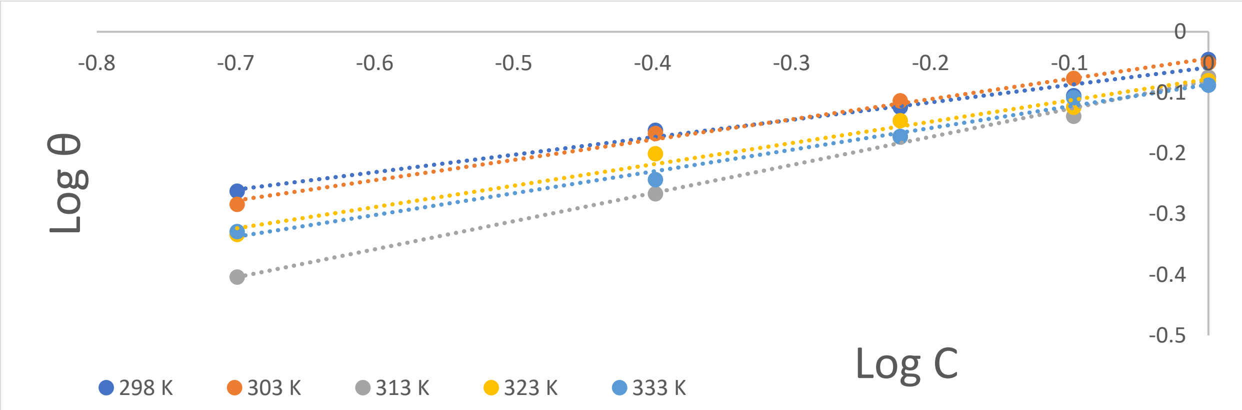

Figure 4: Freundlich isotherm plot for chlorpromazine on mild steel surface in 0.5M H_2SO_4 solutions at various temperature for 1 hour

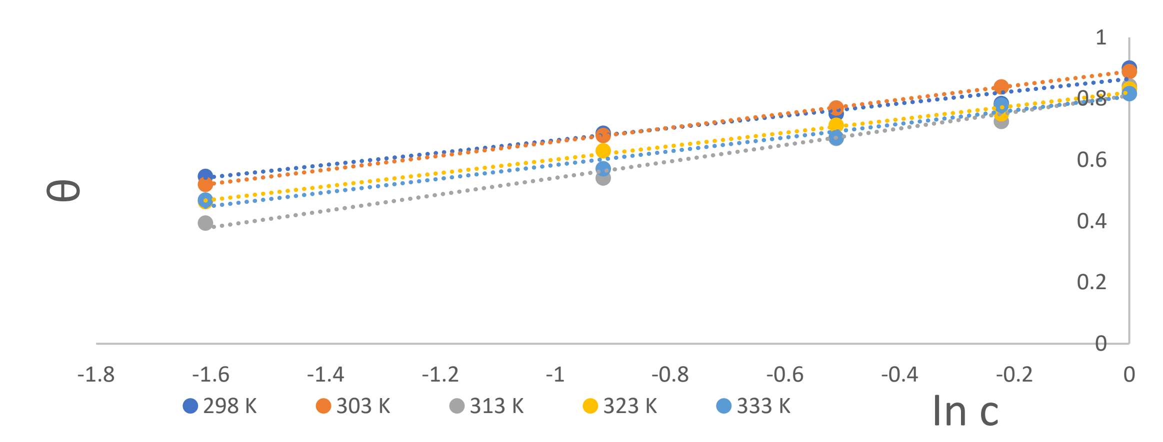

Figure 5: Temkin isotherm plot for chlorpromazine on mild steel surface in 0.5 M H_2SO_4 solutions at various temperature for 1 hour

Table 4: Freundlich parameters for the adsorption of chlorpromazine on mild steel surface 0.5 M H_2SO_4 for 1 hour.

T (K)	InK_{ads} (Kj/mol)	a/n	ΔG_{ads}	R^2
298	0.2874	-0.0588	-10.66	0.9746
303	0.3338	-10.96	-10.96	0.9952
313	0.4627	-3338	-11.66	0.9652
323	0.3511	-0.0779	-11.73	0.9834
333	0.358	-0.087	-12.11	0.9882

Table 5: Temkin parameters for the adsorption of chlorpromazine on mild steel surface 0.5 M H_2SO_4 at 1 hour.

T (K)	InK_{ads}	a/n	ΔG_{ads} (Kj/mol)	R^2
298	0.2006	0.8646	-10.45	0.9594
303	0.2292	0.887	-10.70	0.9999
313	0.2688	0.8101	-11.15	0.98
323	0.2192	0.8204	-11.37	0.9913
333	0.2237	0.8072	-11.74	0.9712

Electrochemical impedance spectroscopy

(Wu *et al*.,2023) Electrochemical experiments were carried out using the traditional three-electrode system by Reference 600 (Gamry Instrument Potentiostat/Galvanostat/ZRA). A graphite electrode (counter), a reference (saturated calomel electrode (SCE)) and mild steel electrode (working) with an exposed area of 3.0 cm^2 was used. The ethanol and acetone were used to clean the polished working electrode and then immersed in the solution for 24 hours to stabilization of the steady-state potential and the measurements have been done at the frequency range of 10^{-1} kHz to 10^4 Hz with using the AC signals of the 5 mV peak-to-peak an amplitude of 5 mV at open circuit potential (OCP) at Bode plot and fitting plot. Zview Analyst 4.5 Software was used for plotting and fitting data. Each experiment was repeated at least two times to check the reproducibility. All tests have been performed in dearated solutions under unstirred conditions at 298 K (Ebenso *et al*., 2021).

At NYQUIEST PLOT, big semi- circle indicate the high resistance to corrosion while the small semi-circle can be prone to corrosion, the higher the concentration of the inhibitor, the lower the corrosion rate. At BODE PLOT, the higher the concentration, the lower the corrosion rate, this also give good results as the chlorpromazine can inhibit corrosion in acid medium. The Nyquist diagram of mild steel 0.5 M H_2SO_4 and with some concentrations of expired chlorpromazine at 333 K are displayed in Figs 6 and fig 8.

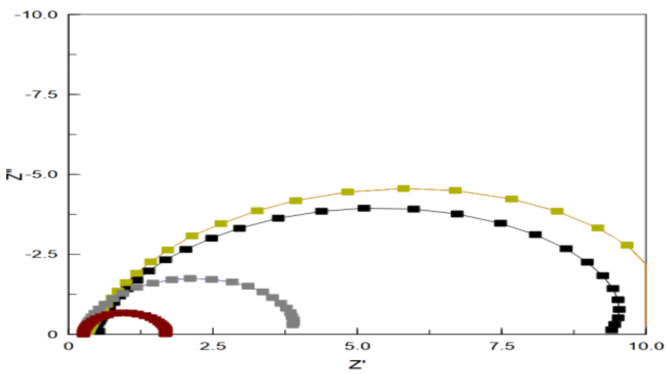

Figure 6: Nyquest plots of mild steel immersed in 0.5 M H_2SO_4 static solution without and with inhibitor

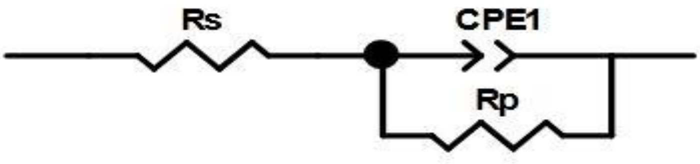

Figure 7: Equivalent circuit model used to analyze electrochemical impedance spectroscopy data

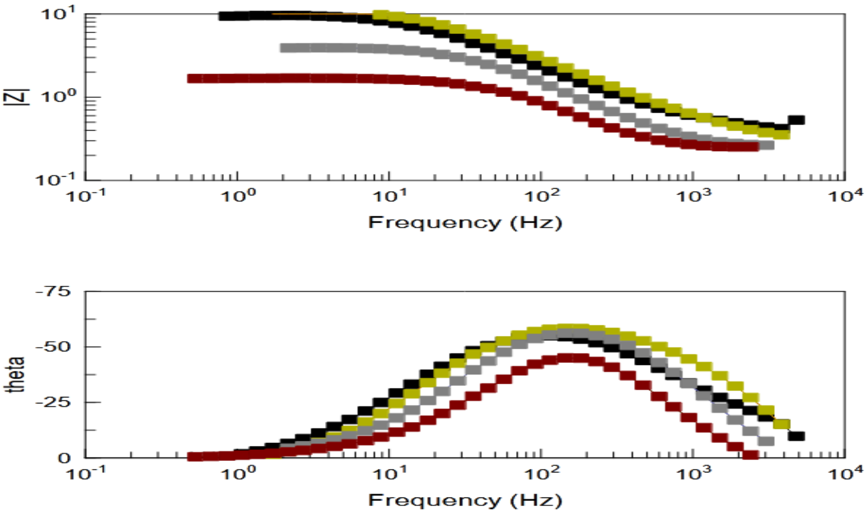

Figure 8: Bode plots of mild steel immersed in 0.5 M H_2SO_4 static solution without and with inhibitor

It is evident from this fig 6 and fig 8, the EIS diagram shows semicircle at fig 6. The deviation of ideal behavior owing to the frequency dispersion as a result of the coarseness and heterogeneity of the mild steel surface. As the concentricity of the expired chlorpromazine increases, the radius and size of semicircles increases. This demonstrate that the characteristics of the coating film on the surface of the mild steel increased. From the Bode diagram (Fig 7), the phase angle vs. frequency curves of the bare electrode conformed to a one-time constant model.
In order to fit the EIS data, all the data were fitted using the appropriate equivalent circuit as given in the insert, in which Rs is the solution resistance, Rct is the charge transfer resistance, Cdl is the double-layer capacitance. Table 6 lists the fitting results of electrochemical impedance spectroscopy; the Chi-Squared values were between 10^{-1} and 10^4, which suggests that the fitting results are reliable. With the increase in pH, both Rct and Rp showed a rising tendency. Generally, the Rct of the electrode is inversely proportional to the corrosion rate and can be used to characterize the corrosion rate. The EIS results were in good agreement with the results of the polarization curves.

Table 6: EIS data of Mild steel in 0.5 M H_2S0_4 and in the presence of different concentrations of Chlorpromazine at 298K for 24 hours

Conc. (g)	Rs Ω m^2	Rp Ω m^2	Rct Ω m^2	CPE µF cm^{-2}	θ	%IE
Blank	0.24954	1.431	0.0640	998.14	_	_
0.2	0.25408	3.757	0.1746	543.36	0.6334	63.34
0.4	0.3674	6.843	0.2977	292.47	0.7850	78.50
0.6	0.4215	9.819	0.3630	225.17	0.8237	82.37
0.8	0.4075	10.48	0.3548	416.61	0.8196	81.96
1.0	0.3908	168.80	0.3878	436.73	0.8350	83.50

CONCLUSION

It has been confirmed in this study that the rate of corrosion decreases with increase in concentrations of inhibitor. The corrosion inhibition efficiency of the chlorpromazine increased with increase in concentration, which implies that corrosion rate was found to decrease with increase in concentration of inhibitor. The adsorptions of the chlorpromazine on the mild steel surface in 0.5 M H_2SO_4 solution obey Langmuir and Freundlich isotherm. Chlorpromazine in the acid media increases the activation energy of corrosion process which indicates physical adsorption. Thermodynamics of the inhibitor adsorption is in accordance with spontaneous adsorption that supports physiosorption mechanism while the heat of the reaction supports endothermic process with increasing degree of association.

From the electrochemical study, a mixed type corrosion inhibitor exists between the inhibitor and the metal. Electrochemical tests have demonstrated that the charge transfer process controls the corrosion reaction. Electrochemical impedance spectroscopy (EIS) techniques displayed that an increase in the chlorpromazine concentration increases Rp on the mild steel surface and decreases CPE

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