

INHIBITION OF MILD STEEL CORROSION IN ACID MEDIUM

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(Received: February 2017 / Revised: May 2017 / Accepted: September 2017)

ABSTRACT

Since inhibition is the simplest mechanism used for mitigating the corrosion of metals and alloys, particularly in acidic environments, the present work aims to investigate the inhibiting effect of N-benzyl-N'-phenyl thiourea (BPTU) and N-cyclohexyl-N'-phenyl thiourea (CPTU) on mild steel corrosion in 0.1M HCl medium using the Tafel extrapolation technique. Tafel experiments were conducted with ± 250 mV vs. rest potential (RP) in steps of 20 mV from the cathodic side for recording the corrosion currents, and then, the Tafel plot of potential vs. current was drawn for determining the corrosion current density (i_{corr}). The linear polarization method was also used for validating the Tafel results. It was performed by polarizing the specimen with ± 20 mV vs. RP in steps of 5 mV, and the corrosion currents were noted. The plot of potential vs. current was drawn for calculating i_{corr} . The study reveals that both BPTU and CPTU act as anodic inhibitors for mild steel in the HCl medium, and good inhibition efficiency ($>97\%$) was evidenced from both the compounds even at elevated temperatures. The study also reveals that the investigated compounds get adsorbed quickly on the steel surface, following Temkin's adsorption isotherm. The kinetic parameters obtained from the study indicated that the inhibition was governed by a chemisorption mechanism and the presence of inhibitors substantially reduced the metal dissolution in the studied temperature range. The investigation shows that there was a good correlation between the Tafel extrapolation and linear polarization results.

Keywords: Acid pickling; Activation energy; Hydrochloric acid; Inhibition of mild steel corrosion; Polarization

1. INTRODUCTION

It is well-known that the use of organic compounds as inhibitors is a simple approach for mitigating the corrosion of metals and alloys in an acid medium (Bentiss et al., 2000). The presence of these organic compounds in the acid media reduces the rate of electrochemical reactions by forming an adsorption layer on the metal surface and thereby blocking the reaction sites (Govindaraju et al., 2009; Anand & Balasubramanian, 2011; Zerga et al., 2009; Mora-Mendoza et al., 2002). The majority of organic compounds used as inhibitors in acid media contain hetero atoms, such as N, S, and O, through which the compounds are quickly adsorbed on the surface of metals and alloys and thereby retard the anodic dissolution of metals and alloys.

Amongst the various inhibitors, derivatives of thiourea have been widely used for inhibiting the corrosion of various metals and alloys in acid media (Quartarone et al., 2012; Pavithra et al.,

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Permalink/DOI: <https://doi.org/10.14716/ijtech.v8i5.873>

2010). The inhibition action of N-(furfuryl)-N'-phenyl thiourea (FPTU) on the corrosion of mild steel in HCl medium was studied using the potentiodynamic polarization technique (Divakara et al., 2007). The study revealed that good inhibition efficiency (>97%) was achieved even in very low concentration of FPTU and the molecules of FPTU was chemically adsorbed on the steel surface. The study on the corrosion inhibition of 304 SS in HCl solution by N-(furfuryl)-N'-phenyl thiourea (FPTU) was carried out using potentiodynamic polarization methods at different temperatures and inhibitor concentrations (Herle et al., 2011). The investigation showed that FPTU worked as an efficient anodic inhibitor and the adsorption of the compound obeys Temkin's adsorption isotherm. The inhibition of the corrosion of stainless steel (304 SS) by N-(2-mercaptophenyl)-N'-phenyl thiourea (MPTU) was studied using different techniques, such as weight loss, potentiodynamic polarization and impedance spectroscopy (Ramdev et al., 2013). The study demonstrated that MPTU was a mixed inhibitor that yielded protection efficiency more than 90%. Dicyclohexyl thiourea (DCTU) was used as an inhibitor for inhibiting the corrosion of stainless steel (304L) in 2 M HCl medium and found that DCTU acts as an excellent anodic inhibitor for the corrosion of steel. Good protective efficiency of 90% was observed in HCl medium (Shen, 2011).

The choice of suitable organic compounds as corrosion inhibitors primarily depends on the type of acid and its concentration, solution temperature, and the kind of metal or alloy exposed in the acidic medium (Shetty et al., 2007). Since mild steel has wide applications in various industries owing to its easy availability, low cost, and uncomplicated fabrication method, the assessment of corrosion behavior of mild steel in various corrosive environments has received a significant amount of interest (Ostovari et al., 2009). HCl is known to be the working horse in the pre-treatment of mild steel components and is normally used for acid pickling and de-scaling, and acidizing of oil and gas wells (Emregul & Hayvali, 2006).

The following are the reasons for selecting and synthesizing the novel thiourea derivatives, such as BPTU and CPTU, for the corrosion of mild steel in HCl medium: (a) From the literature, it is observed that thiourea derivatives are the most effective inhibitors for ensuring excellent protection to the metals and alloys against corrosion in acid media; (b) In general, acid pickling and descaling operations are carried out at elevated temperature. The addition of inhibitor to the acidic medium helps to prevent the further dissolution of metals by forming a thin protective film on the surface of the metal and thereby preventing the metal - media interaction. It has been observed from the previous studies (Shetty et al., 2005; Shetty et al., 2008) that both BPTU and CPTU exhibit excellent performance. This motivates the authors to further investigate the effect of these compounds on the corrosion of mild steel in HCl medium at different temperatures up to 60°C. The experiments were also conducted at lower temperatures for determining the thermodynamic parameters to determine the kind and nature of adsorption; (c) The selection of these compounds as inhibitors for the corrosion of mild steel in acid medium is also attributed to the fact that the method used for synthesizing these compounds is simple, cost-effective and effective at forming an insoluble stable film on the mild steel surface owing to the presence of S and N atoms in their molecular structures. The main focus this study is to determine how these derivatives of thiourea maintain their stability and protection ability against metallic corrosion at higher temperatures in HCl medium.

2. MATERIALS AND METHODS

2.1. Specimen Preparation

The mild steel specimen with an exposed area 0.786 cm² and the chemical composition (wt %): C: 0.205; Si: 0.06; Mn: 0.55; S: 0.047; P: 0.039; and balance Fe was used in this study. The test sample was mirror polished with emery papers, cleaned with distilled water, and dried in air.

2.2. Inhibitors Preparation

The compounds BPTU and CPTU were synthesized according to the procedure of synthesizing the similar compounds (Moore & Crossly, 2003). The BPTU is the condensation product of benzyl amine and phenyl-isothiocyanate whereas, CPTU is the condensation product of cyclohexyl amine and phenyl-isothiocyanate and the re-crystallization of these compounds were done by ethanol. The purity of the compounds was tested by elemental analysis and the melting point values shown in Tables 1 & 2, respectively. The molecular structures of the compounds are shown in Figure 1.

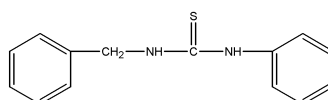
Table 1 Elemental analysis of the compounds studied

Inhibitors	Molecular formula	Calculated (found)%				
		C	H	N	O	S
BPTU	C ₁₄ H ₁₄ N ₂ S	69.39	5.82	11.56	-	13.23
		(69.26)	(5.79)	(11.60)	-	(13.19)
CPTU	C ₁₃ H ₁₈ N ₂ S	66.63	7.74	11.95	-	13.68
		(66.50)	(7.71)	(11.90)	-	(13.62)

Table 2 Physical properties of inhibitors studied

Inhibitors	Molecular weight	Melting point (°C)
BPTU	242.34	158
CPTU	234.37	149

N-benzyl-*N'*-phenyl thiourea



N-cyclohexyl-*N'*-phenyl thiourea

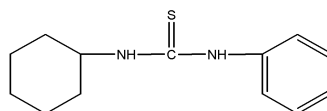


Figure 1 Structural formula of thiourea derivatives

2.3. Tafel Extrapolation Study

Tafel extrapolation studies were carried out by using a Wenking potentiostat (LB95L), and the electrochemical cell, which contains three electrodes (platinum electrode used as auxiliary electrode, saturated calomel electrode used as reference electrode and the mild steel specimen is used as working electrode), as shown in Figure 2.

The steady state rest potential (RP) was noted after about 25-30 min. and then, Tafel experiments were carried out at 30°C with ± 250 mV vs. RP in steps of 20 mV/min and the corrosion currents (I) were noted in the absence and presence of different concentrations (0.0001-0.0004 mol/L) of inhibitors. The plot of potential vs. log I was drawn for different concentrations of BPTU and CPTU for determining the corrosion current density (i_{corr}) and the corrosion potential (E_{corr}). The corrosion current (I_{corr}) and the corrosion potential (E_{corr}) were obtained by extrapolating the Tafel regions (straight line regions) of both anodic and cathodic

curves shown in the schematic diagram (Figure 3). The experiments were repeated for 45 and 60°C in the absence and presence of 0.0001, 0.0002, 0.0003, and 0.0004 mol/L of inhibitors.



Figure 2 Experimental set up

The corrosion rate was calculated using Equation 1, and the linear polarization method was used for validating the Tafel results obtained for the corrosion of mild steel in HCl medium.

$$\text{Corrosion rate (CR), mpy} = \frac{0.129 \times \text{Eq. Wt} \times i_{\text{corr}}}{D} \quad (1)$$

where, i_{corr} has units in $\mu\text{A}/\text{cm}^2$, Eq. wt is the equivalent weight of the specimen taken as 27.925 g, and D is the density of the specimen taken as 7.86 g/cc.

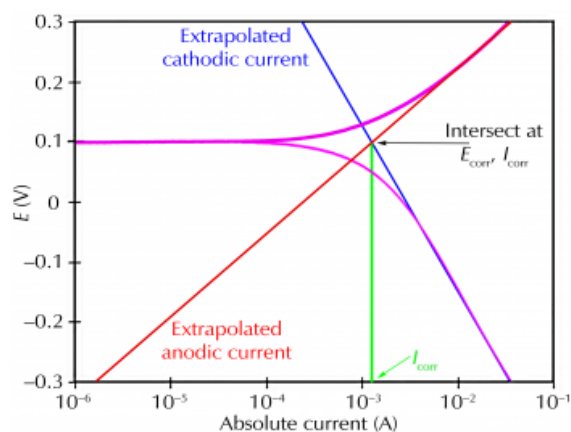


Figure 3 Schematic representation of Tafel plot

2.4. Linear Polarization Study

The linear polarization study was performed from ± 20 mV vs. RP with a scanning rate of 5 mV/min, and the corresponding steady state corrosion current (I) was noted for uninhibited (blank) and inhibited system for different temperatures. The plot of potential (E) vs. I was drawn, and the slope of the E vs. I curve was used for determining i_{corr} , which is calculated as follows:

$$i_{\text{corr}} = 0.026/\text{slope} \quad (2)$$

Figure 4 illustrates the schematic representation of the linear polarization curve and the slope of E vs. I is taken in the straight line region of the curve for calculating the i_{corr} values.

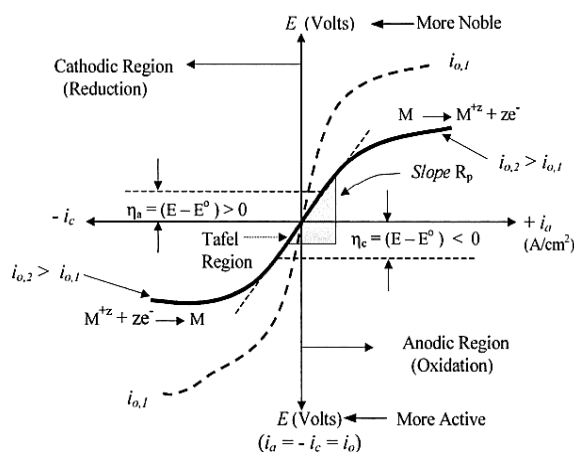


Figure 4 Schematic representation of linear polarization curve

The percentage inhibition efficiency (% IE) and the degree of surface coverage (θ) for the corrosion of mild steel were calculated by using the following relations;

$$\% IE = \left[\frac{(i_{corr} - i_{corr(inh)})}{i_{corr}} \right] \times 100 \quad (3)$$

where, i_{corr} and $i_{corr(inh)}$ are the current densities in the nonexistence and existence of inhibitors, respectively.

$$\theta = \left[\frac{(i_{corr} - i_{corr(inh)})}{i_{corr}} \right] \quad (4)$$

3. RESULTS AND DISCUSSION

The results of the Tafel extrapolation studies carried out in the presence and absence of BPTU and CPTU for the corrosion of mild steel in 0.1M HCl solution at different temperatures are presented in Table 3.

Table 3 Tafel extrapolation results for mild steel corrosion in 0.1M HCl

C (mol/L)	30°C			45°C			60°C		
	E_{corr} (mV)	CR (mpy)	IE (%)	E_{corr} (mV)	CR (mpy)	IE (%)	E_{corr} (mV)	CR (mpy)	IE (%)
Blank	-540	298.46	-	-535	495.04	-	-530	674.94	-
BPTU									
0.0001	-495	4.51	98.49	-483	12.21	97.53	-502	22.51	96.66
0.0002	-490	3.75	98.74	-482	10.99	97.78	-493	20.49	96.96
0.0003	-485	3.32	98.89	-476	10.26	97.93	-490	19.75	97.07
0.0004	-478	3.45	98.84	-470	10.39	97.90	-482	20.36	96.98
CPTU									
0.0001	-485	4.72	98.42	-488	12.28	97.52	-495	22.85	96.61
0.0002	-474	4.15	98.61	-478	11.24	97.73	-484	20.76	96.69
0.0003	-470	3.98	98.87	-470	10.50	97.88	-475	19.98	97.04
0.0004	-469	4.25	98.58	-463	10.69	97.84	-468	20.67	96.94

3.1. Effect of Inhibitors on Mild Steel Corrosion

The Tafel plot for mild steel in 0.1 M HCl in the absence and presence of 0.0004 mol/L of both BPTU and CPTU at 30°C is shown as an example in Figure 5. From Figure 5, it can be observed that the Tafel curves of both BPTU and CPTU were shifted to the left compared to the Tafel curve shown for the blank (uninhibited system). This demonstrates that there is a noticeable reduction in corrosion current in the presence of 0.0004 mol/L of inhibitors at 30°C. The decrease in corrosion current in the presence of inhibitors at different temperatures shown in Table 5 is also a clear evidence for the effectiveness of both the compounds in mitigating the corrosion of mild steel in HCl solution. From Figure 5, it can also be observed that the corrosion potential (E_{corr}) swings in the positive direction or the anodic direction by shifting the Tafel curves of both the inhibitors in the upward direction. This swing in E_{corr} in the positive region indicates that the investigated compounds predominantly act as anodic inhibitors for mild steel in HCl medium (Divakara et al., 2007). Similar trends were noticed in 0.0004 mol/L of inhibitors concentration at 45 and 60°C and also in 0.0001, 0.0002, and 0.0003 mol/L of inhibitors at different temperatures. Shift in the polarization curves (shown in Figure 5) to the left or to the lower corrosion current values in the presence of 0.0004 mol/L of inhibitors, indicating that the dissolution of the metal was reduced to a greater extent in HCl medium.

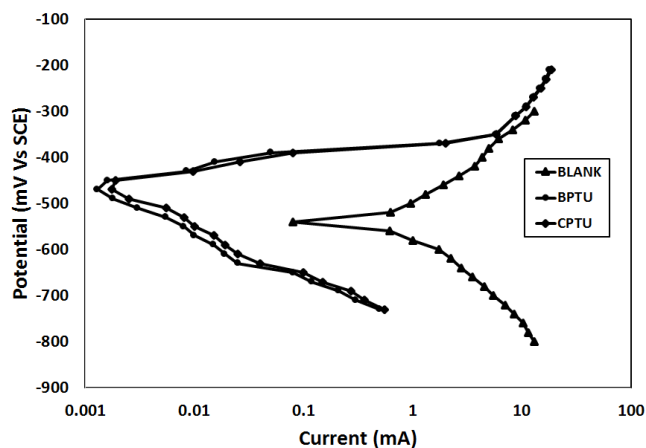


Figure 5 Tafel extrapolation plot for mild steel in 0.1 M HCl with and without the presence of 0.0004 mol/L of inhibitors at 30°C

The investigation shows that the inhibition efficiency (IE) has a positive correlation with the concentrations of inhibitors, except in 0.0004 mol/L in the range of temperatures studied. The addition of 0.0004 mol/L of inhibitors to the corrosive medium decreases the IE of the compounds at all temperatures studied. This indicates that the optimal concentration of the inhibitors required to achieve maximum inhibition efficiencies at different temperatures is 0.0003 mol/L. This increase in IE may be attributed to the creation of adsorption film on the surface of the mild steel which reduces the effective area of attack. The decrease in efficiency for 0.0004 mol/L of inhibitors may be attributed to the desorption of the inhibitor molecules from the steel surface. Owing to this, the rate of metal dissolution or the corrosion rate starts increasing and thus decreasing the inhibition efficiency and the degree of surface coverage values. The study also reveals that 0.0003 mol/L of BPTU and CPTU can be efficiently used as inhibitors for mild steel corrosion in HCl medium in industries. The linear polarization results obtained for the corrosion of mild steel in the presence and absence of both BPTU and CPTU at different temperatures in 0.1M HCl solution are presented in Table 4. From Table 3 and Table 4, it can be observed that the Tafel extrapolation and linear polarization results closely match each other. This confirms the accuracy of the Tafel extrapolation results obtained for corrosion of mild steel in HCl medium.

Table 4 Linear polarization results for mild steel corrosion in 0.1M HCl

C (mol/L)	30°C			45°C			60°C		
	OCP (mV)	CR (mpy)	IE (%)	OCP (mV)	CR (mpy)	IE (%)	OCP (mV)	CR (mpy)	IE (%)
Blank	-555	295.68	-	-540	480.52	-	-535	670.50	-
BPTU									
0.0001	-500	4.25	98.56	-480	11.10	97.69	-500	22.54	96.64
0.0002	-490	3.76	98.73	-480	10.00	97.92	-495	20.48	96.95
0.0003	-485	3.45	98.88	-470	9.71	97.98	-490	19.80	97.05
0.0004	-480	3.49	98.82	-465	9.90	97.94	-480	20.25	96.98
CPTU									
0.0001	-495	4.75	98.39	-490	11.77	97.55	-495	22.78	96.60
0.0002	-480	4.10	98.61	-485	10.77	97.76	-490	20.81	96.90
0.0003	-475	3.97	98.66	-480	10.00	97.92	-485	19.96	97.02
0.0004	-480	4.23	98.57	-470	10.19	97.88	-480	20.48	96.95

3.2. Influence of Temperatures on % IE and Corrosion Rate

The variation in the % IE values with solution temperatures for different concentrations of BPTU and CPTU is shown in Tables 3 and 4. From Tables 3 and 4, it can be seen that the % IE of the investigated compounds decreases marginally with an increase in the solution temperature from 30 to 60°C. This indicates that these compounds were insensitive to the range of temperatures studied. The highest IE values exhibited by both the compounds in the critical or optimal concentration (0.0003 mol/L) at 30, 45, and 60°C were 98.87%, 97.93% and 97.04%, respectively. This confirms that both BPTU and CPTU did not lose their inhibiting ability at elevated temperatures for the corrosion of mild steel in HCl solution.

The good performance exhibited by these compounds may be attributed to the existence of the N and S atoms in the compounds, which results in their quick adsorption on the surface of the metal, thus forming an insoluble unvarying film on the mild steel surface. The S atoms present in both BPTU and CPTU have a permanent negative charge and N atoms present in these compounds have a positive charge. As the molecules of these compounds approach the metal surface, the electric field of the double layer enhances the polarization of molecules and induces extra charges on both the S and N atoms. Owing to this condition, the adsorption of inhibitor molecules on the steel surface increases. Thus, both compounds exhibited excellent performance against the corrosion of mild steel in HCl medium (Shetty et al., 2016).

Further, the tendency to form a co-ordinate bond and the degree of inhibition can be increased by enhancing the effective electron density at the function group of the organic compounds. Higher inhibition efficiencies exhibited by both BPTU and CPTU even at the elevated temperature may be attributed to their adsorption on the mild steel surface through the polar groups and π -electrons on the aromatic ring (Selvi et al., 2003). This leads to larger coverage of the mild steel surface by the molecules of the compounds, thus ensuing in higher inhibition efficiency from both compounds. Since the S atom is less electronegative than N and has two electron pairs available in the active site for co-ordination, the bonding among the molecules of inhibitors and the steel surface possibly occurs through the S atom. The influence of temperature on the corrosion rate of mild steel in the absence and presence of inhibitors is shown in Tables 3 and 4. From Tables 3 and 4, it can be observed that there is a direct correlation between the solution temperature and corrosion rate. This may be due to an increase in conductance of the aqueous medium, which accelerates the anodic dissolution of metals with an increase in temperature.

3.3. Kinetic Parameters of Adsorption

The mechanism of adsorption of inhibitors onto the metal surface may be acknowledged by determining the values of the kinetic parameters of adsorption. The free energy of adsorption (ΔG_{ads}) and the activation energy (E_a) were determined with the help of following relationships (Quaroishi & Sardar, 2004),

$$\Delta G_{\text{ads}} = -RT \ln (55.5K) \quad (5)$$

where, R is the universal gas constant in J/k/mol, T is the temperature in Kelvin, 55.5 is the concentration of water in mol/L, and K is the equilibrium constant.

$$K = \theta / C (1-\theta) \quad (6)$$

where, θ is the degree of surface coverage on the metal surface, and C is the inhibitor concentration in mol /L.

$$\ln (r_2/ r_1) = -E_a \Delta T/ (R \times T_2 \times T_1) \quad (7)$$

where, r_1 and r_2 are the corrosion rates at temperatures T_1 and T_2 , respectively, and ΔT is the difference in temperature (T_1-T_2).

The ΔG_{ads} values for the compounds studied were more than 40 kJ/mol (Table 5), demonstrating that BPTU and CPTU were chemically adsorbed on the metal surface in HCl medium (Quraishi et al., 2002). The chemical adsorption or chemisorption can also be confirmed from the effect of inhibitor concentrations on the corrosion potential (E_{corr}). The study shows that the presence of BPTU and CPTU shifts the E_{corr} in the positive direction. This indicates the control of the anodic reaction owing to the blocking of anodic site by chemisorption of the electron rich N-S function ability to the positive centers of the metal (Ali et al., 2005). The negative values of ΔG_{ads} indicated impulsive adsorption and a strong interface between the inhibitor molecules and the surface of the metal (Savithri & Mayanna, 1996). Table 5 shows that E_a values in the presence inhibitors were higher than that in the case of the uninhibited solution. This indicates that the disbanding of the metal is retarded in the existence of inhibitors and the compounds studied were also more efficient at lower temperatures (Vishwanatham & Emranuzzaman, 1998; Quraishi & Sharma, 2004).

Table 5 E_a and ΔG_{ads} for mild steel in 0.1 M HCl and 0.0004 mol/L of inhibitors

System	E_a (kJ/mol)	- ΔG_{ads} (kJ/mol)		
		30°C	45°C	60°C
0.1 M HCl	25.33	-	-	-
BPTU	54.48	41.03	41.47	42.40
CPTU	46.75	40.52	41.39	42.36

Temkin's adsorption plot for mild steel in the presence of 0.0001–0.0004 mol/L of BPTU and CPTU at different temperatures are shown in Figures 6a, 6b. It can be seen that θ has a linear correlation with $\log C$. This signifies that the compounds adsorbed on the steel surface follows Temkin's adsorption isotherm and its applicability verifies the assumption of a single layer adsorption on a uniform homogeneous metal surface (Stoyanova et al., 1997; Shetty et al., 2007).

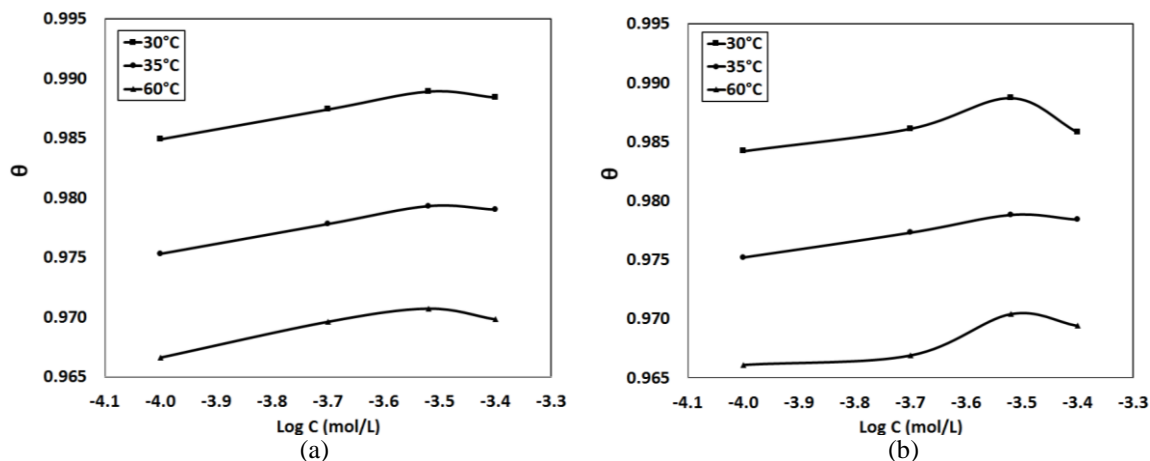


Figure 6 Temkin's adsorption plot for mild steel in 0.1 M HCl at different temperatures for: (a) BPTU; and (b) CPTU

4. CONCLUSION

The polarization study reveals that the compounds investigated act as excellent anodic inhibitors for mild steel in HCl solution. The study also reveals that the presence of BPTU and CPTU enhanced the corrosion resistance of mild steel. The adsorption of these compounds on the mild steel surface follows Temkin's adsorption isotherm, and the inhibition was governed by a chemisorption mechanism. The study results demonstrated that the IE values of the compounds were nearly constant in the range of temperatures and inhibitors concentrations studied. The exceptionally good performance demonstrated by BPTU and CPTU, even at very low concentrations and at elevated temperatures, confirmed that these compounds can be efficiently used as inhibitors for mild steel corrosion in HCl solution.

5. ACKNOWLEDGEMENT

The authors would like to thank Manipal University, Manipal, India, for providing facilities for conducting experiments.

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