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RESEARCH ARTICLE

CORROSION INHIBITION OF MILD STEEL WITH AQUEOUS EXTRACT OF *CALOTROPIS PROCERA L.* LEAVES IN 1M HCl SOLUTION

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ABSTRACT

The inhibition of mild steel (MS) in 1 M HCl solution with aqueous extract of *Calotropis procera L.* leaves (AECPL) was studied by weight loss method at 303-333K temperatures. It was found that inhibition efficiency increased with increase in concentration of extract and decreased with increase in temperature. Maximum 60.86% inhibition efficiency was observed at 303 K and at 8% (v/v) concentration of extract. Adsorption of extract at mild steel surface follows Langmuir adsorption isotherm. Physiosorption is proposed by the values of Gibbs free energy, variation in inhibition energy with temperature and with activation energy values trend. Negative Gibbs energy reveals the spontaneity of inhibition process in extract at studied temperatures.

INTRODUCTION

Steel corrosion inhibition phenomenon have become an industrial and academic topic especially in acidic media (Bentiss et al., 2000). This is because of the increasing industrial applications of acid solutions in acid pickling, industrial cleaning, acid descaling, oil-well acidizing in oil recovery and the petrochemical processes. Besides this corrosion rates are very high in aqueous acidic media especially when soluble corrosion products are formed (Raja et al., 2013). Therefore exploring better green corrosion inhibitors are important for its practical applications and environmental friendly point of view. Synthetic organic corrosion inhibitors are effective but costly, environmental polluting and harmful to living beings during their manufacture and applications. So currently researches on corrosion are focused on "green corrosion inhibitors" that show good inhibition efficiency with low risk of environmental pollution (Amin et al., 2007) All plant extracts, which have been shown effective corrosion inhibitors are comparatively cheap, easily available, biodegradable and called as green corrosion inhibitors.

Different plant parts have been studied as corrosion inhibitors in acidic and alkaline media (Ostovari et al., 2009; Alaneme et al., 2015; Kamal et al., 2012; James et al., 2009; Ambrish et al., 2010; Ibrahim et al., 2011; Bhardwaj et al., 2015). In the present study aqueous extract of leaves of *Calotropis Procera L.* has been chosen as corrosion inhibitor for mild steel in 1M HCl solution and findings are discussed in the light of inhibitor concentration, temperature, adsorption properties, kinetic properties, thermodynamic properties. Weight loss (gravimetric analysis) method is applied to obtain inhibition efficiencies.

MATERIALS AND METHODS

Preparation of extract

The leaves of *Calotropis procera L.* plant were taken, washed and air dried for 6-7 days, crushed and grind mechanically. 20 g of ground leaves were heated in 250 ml distill water for one hour using air condenser at 70°C - 80°C. This extract was left overnight and then filtered and make up to 250 ml with distill water for the experiment.

Preparation of steel specimens

Cylindrical mild steel specimens of 4.9 cm length and 0.70 cm diameter were taken and abraded with a series of emery papers,

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degreased with acetone, washed with distill water, dried and constant weight was recorded by electronic balance.

Solution Preparation

1M HCl solution was prepared by 37% HCl (Merk Ltd.) using distill water. The employed concentration range of aqueous extract of *Calotropis procera* L. leaves (AECL) was 1% to 8% (v/v).

Gravimetric Measurements

Gravimetric method is widely used method because of its reliability and simplicity in corrosion inhibition experiments. For each experiment 100 ml test solutions were taken in 250 ml beaker and cylindrical specimen was immersed in it with plastic thread for one hour. The experiments were carried out at different temperatures ranges from 303 K to 333K in thermostatic water bath. After one hour specimens were removed, washed with distill water, acetone dried and abraded with series of emery papers and then weighted accurately with electronic balance. It was noted that the surfaces of specimens became more rough in test solutions without the inhibitor than the surfaces of specimens which were immersed in test solutions containing different concentrations of inhibitor.

RESULTS AND DISCUSSION

Effect of temperature on corrosion rates

Corrosion rates were calculated by following equation (Mourya et al., 2014; Yadav et al., 2014).

$$C R (\text{g cm}^{-2}\text{min}^{-1}) = \left(\frac{W_1 - W_2}{A t} \right) \quad (1)$$

Where CR is corrosion rate, W_1 is weight loss of mild steel specimen without inhibitor and W_2 is weight loss of mild steel specimen with inhibitor, A is area of MS specimen and t is immersion time.

Table 1 represents that corrosion rates increases with increase in temperatures and decreases with increase in concentration of inhibitor. It is clear that corrosion rates obey Arrhenius type reactions as it increases with increase in solution temperature (31).

Effect of temperature on inhibition efficiency

From the obtained corrosion rates, inhibition efficiencies were calculated by using following equation (Mourya et al., 2014; Behpour et al., 2011).

$$IE \% = \left(\frac{CR - CR_{inh}}{CR} \right) \times 100 \quad (2)$$

Where CR is the corrosion rate in absence of inhibitor and CR_{inh} is corrosion rate in presence of inhibitor. AECL are given in Table 1.

Data in Table 1 reveal that inhibition efficiency increased with an increase in inhibitor concentration. This suggests that the

inhibitor species adsorbed on steel /solution interface where the adsorbed species mechanically screen the coated part of steel surface from the action of corrosion medium (31). The variation in inhibition efficiency was detected with increasing temperatures in Table 1.

It is clear from the data that inhibition efficiency decreases with rise in temperatures, suggests possible desorption of adsorbed species. It further suggests physisorption of inhibitor molecules on steel surfaces (39). The surface coverage θ is calculated by in the given equation (Yadav et al., 2014).

$$\theta = \left(\frac{CR - CR_{inh}}{CR} \right) \quad (3)$$

θ is the surface coverage, CR is the corrosion rate in absence of inhibitor and CR_{inh} is corrosion rate in presence of inhibitor.

Kinetic parameters

Assuming that corrosion rates of steel specimens against concentration of inhibitor obeys kinetic relationship as equation (Khamis et al., 2000; Noor 2005).

$$\log CR = \log K + B \log C_{inh} \quad (4)$$

Where K is rate constant and equal to CR when inhibitor concentration is unity. B is reaction constant which is measure of inhibitor effectiveness and C_{inh} is the concentration v/v % (ml/100ml) of AECL.

Fig. 2 represents plot between $\log CR$ and $\log C_{inh}$ values at various studied temperatures. B and K were calculated by slope and intercept of straight lines of the graph.

The obtained results in Table 2 can be discussed as follows (Noor 2007).

- Negative values of B indicates that corrosion rate is inversely proportional to concentration of inhibitor. In other words the corrosion rates decrease with increase in concentration of inhibitor species.
- The high negative values of B reflects good inhibitive property of inhibitor. High negative value of B can be observed as steep slope in graph (Fig.1).
- Value of B is high at lower temperatures, indicates that inhibitive species is more effective at comparatively lower temperatures.
- The increase in K values with increase in temperature, indicating the increase in corrosion rates with temperatures.

Thermodynamic and activation parameters

The thermodynamic and activation parameters like apparent activation energy E_{act} , enthalpy of activation ΔH^* , entropy of activation ΔS^* were calculated for steel dissolution process. Activation energy E_{act} were calculated by following Arrhenius equation (Yadav et al., 2014).

$$\log CR = \log A - \left(\frac{E_{act}}{2.303RT} \right) \quad (5)$$

Table 1. Inhibition efficiencies and corrosion rates of mild steel corrosion in 1MHCl in different concentration of AECL at different temperatures

Conc (v/v)%	CR x 10 ⁻³ (g/cm ² /min)				IE %			
	303 K	313 K	323 K	333 K	303 K	313 K	323 K	333 K
0	0.69	1.37	2.33	3.07	--	--	---	---
1	0.59	1.21	2.17	2.88	14.49	11.67	06.86	06.18
2	0.41	1.08	1.94	2.81	40.57	20.16	16.73	08.46
3	0.38	0.99	1.93	2.74	44.92	27.73	17.16	10.74
5	0.33	0.86	1.86	2.64	52.17	37.22	20.17	14.10
8	0.27	0.72	1.57	2.56	60.86	47.44	32.61	16.43

Table 2. Kinetic parameters and regression coefficients for the corrosion of mild steel in 1M HCl containing AECL at different temperatures

Temp (° K)	Kinetic parameters		
	R ² values	B	K(x10 ⁻³)(g/cm ² /min)
303	0.974	-0.3584	-0.5736
313	0.968	-0.2514	-1.2755
323	0.898	-0.1363	-2.1923
333	0.956	-0.0538	-2.9080

Table 3. Thermodynamic Activation parameters for the corrosion process of mild steel in solution containing different concentrations of AECL in 1 mol HCl solutions

CONC (v/v %)	E _{act} (KJ/mol)	Δ H [‡] (KJ/mol)	Δ S [‡] (J/mol/K)
0	41.26	38.83	-177.64
1	43.20	41.39	-170.56
2	52.93	50.41	-143.63
3	54.91	52.48	-137.55
5	57.86	54.99	--130.48
8	60.93	58.49	-120.69

Table 4. Adsorption parameters obtained from Langmuir adsorption isotherm (A) and Gibbs free energy, entropy and enthalpy for adsorption process of inhibitor at mild steel in 1 M HCl solution in AECL at different studied temperatures

TEMP (K)	R ²	K _{ads}	Δ G (KJ/mol)	Δ H (KJ/mol)	Δ S (J/mol/K)
303	0.887	0.2358	-13.76		-73.23
313	0.998	0.1389	-12.84	-35.95	-73.83
323	0.912	0.0869	-11.99		-74.17
333	0.994	0.0659	-11.59		-73.15

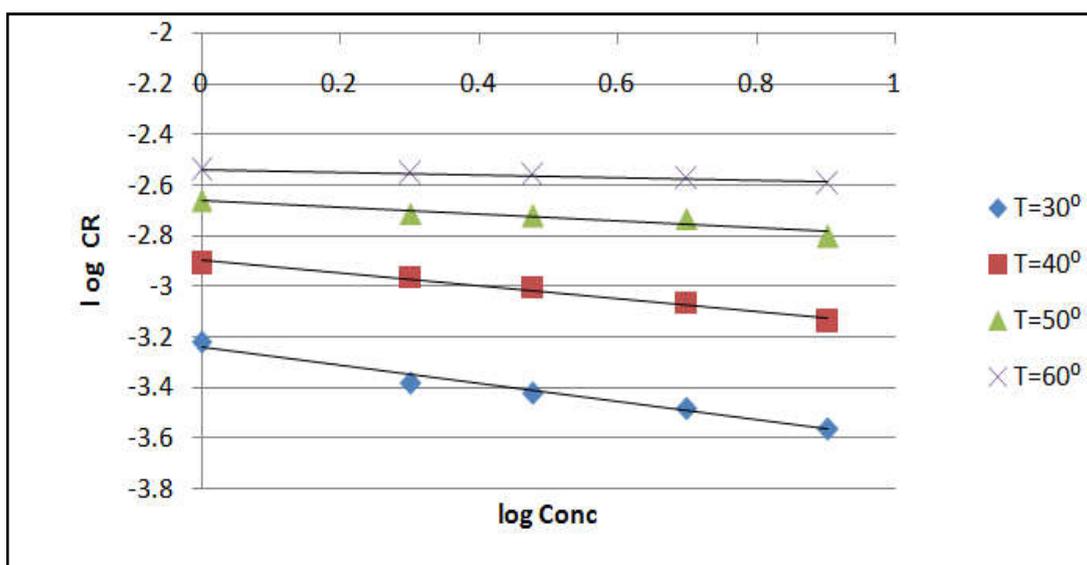


Figure 1. Variation of Log CR vs Log C of corrosion inhibition process by AECL at studied temperatures

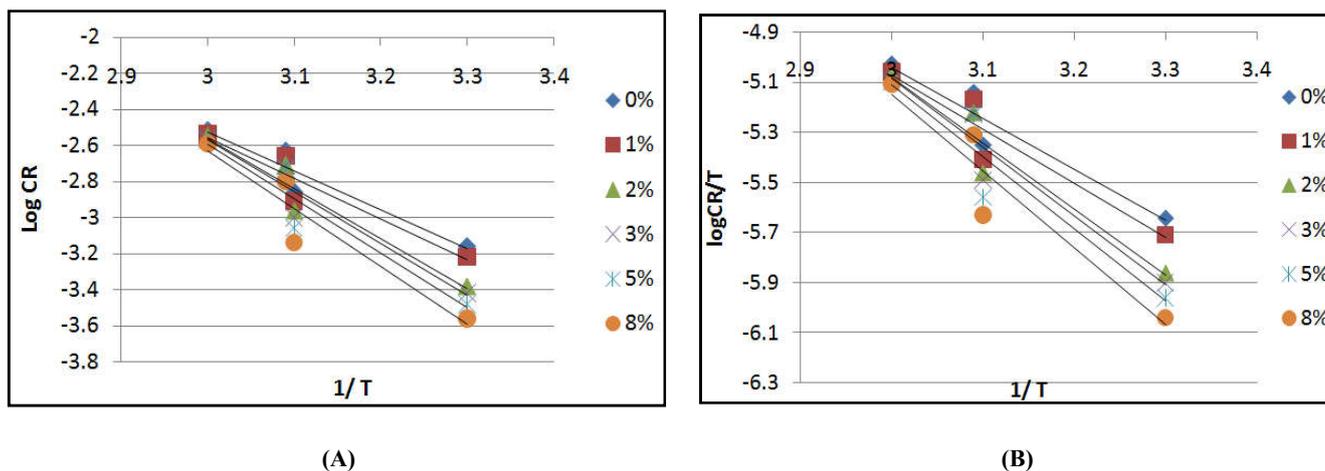


Figure 2. (A) Arrhenius plot and (B) Transition state plot for corrosion of mild steel in 1M HCl in the absence and presence of various concentrations of AECL

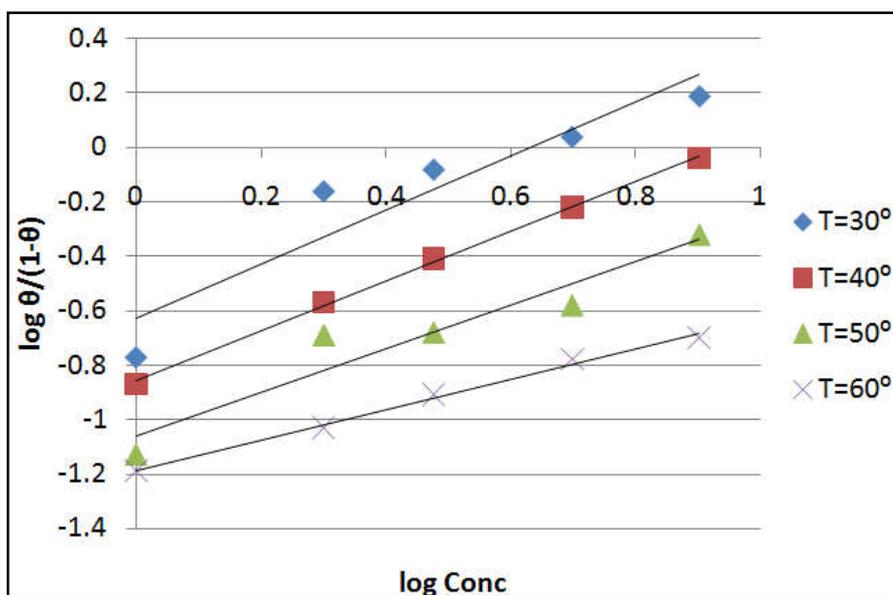


Figure 3. Langmuir adsorption isotherm for corrosion of mild steel in 1M HCl in the absence and presence of various concentrations of AECL at different temperatures

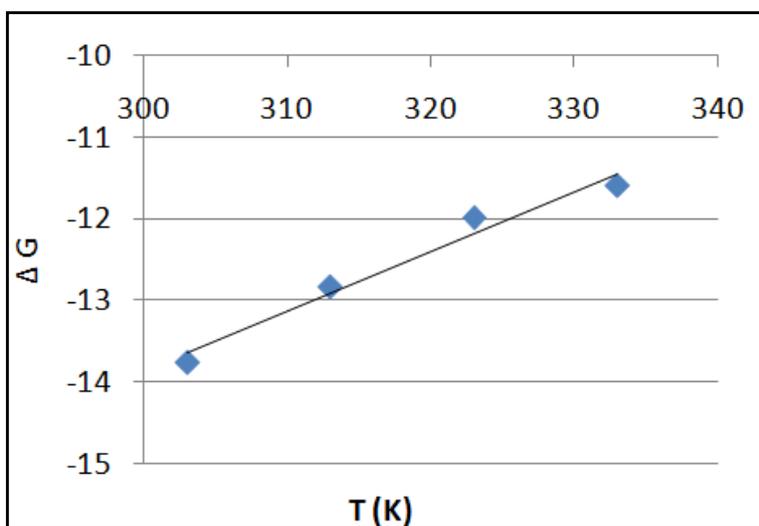


Figure 4. Variation of Gibbs energy change with different studied temperatures

Where A is Arrhenius pre exponential factor, E_{act} is activation energy, R is universal gas constant, T is absolute temperature. The slope of $\log CR$ vs $1/T$ gives the values of activation energies at studied concentrations. Table 3 contains the calculated data of activation energies. The values of activation energies in presence of inhibitor species were found higher than uninhibited solutions. The higher the concentration of inhibitor, the higher the activation energy is observed. It is observed that inhibitive causes a rise in activation energy value when compared to the blank solutions (Manssour *et al.*, 2014). The change in values of activation energies is due to the modification of the mechanism of corrosion process in presence of adsorbed inhibitor molecules (Riggs *et al.*, 1967) and could be often interpreted as an indication for the formation of an adsorbed film on metal surface (Schorr *et al.*, 1972; Clark *et al.*, 1979). The rise in activation energy indicates the formation of energy barrier in corrosion process in presence of the inhibitor and the increase in activation energy with rise in temperature could be interpreted as physical adsorption (40). The values of enthalpy of activation ΔH^* and entropy of activation ΔS^* were calculated by following transition state equation (Yadav *et al.*, 2014).

$$\log\left(\frac{CR}{T}\right) = \left(\log\left(\frac{R}{Nh}\right)\right) + \left[\left(\frac{\Delta S^*}{2.303R}\right) - \left(\frac{\Delta H^*}{2.303RT}\right)\right] \quad (6)$$

Where h is planck's constant, N is Avogadro number R is the gas constant A plot of $\log (CR/T)$ vs $1/T$ gave a straight line with slope of $(-\Delta H^*/2.303R)$ and intercept of $[(\log R/Nh) + (\Delta S^*/2.303R)]$ from which the values of ΔH^* and ΔS^* were calculated. These values are tabulated in Table 3. The values of ΔH^* and ΔS^* in presence of inhibitor were higher than uninhibited solutions. The positive values of ΔH^* reflects endothermic nature of steel dissolution process (Guan *et al.*, 2004) and negative values of ΔS^* indicate the formation of activated complex in rate determining step, represents an association rather than dissociation, means decrease in disorder of system due to adsorption of inhibitor molecules on to the metal surface (Abd-Ei-Nabey *et al.*, 1996). This reveals the formation of an ordered stable layer of inhibitor on steel surface (Yurt *et al.*, 2004).

Adsorption isotherm and gibbs energy

The mechanism of adsorption can also be predicted by metal/electrolyte interface interaction. To understand the nature of adsorption, obtained surface coverage θ were fitted in different adsorption isotherms. The mathematical expressions for Langmuir (Noor and Al-Moubaraki, 2008).

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C_{inh} \text{ (Langmuir isotherm)} \quad (7)$$

Rearranging the above equation

$$(\theta / 1-\theta) = K_{ads} \cdot C_{inh} \quad (8)$$

$$\text{Or } \log (\theta / 1-\theta) = \log K_{ads} + \log C_{inh} \quad (9)$$

Where K_{ads} is the equilibrium constant of adsorption, a is the molecular interaction factor in adsorbed layer, θ is the surface

coverage, $(1-\theta)$ is the uncovered surface, C_{inh} is the concentration of inhibitor

The value of K_{ads} obtained from Langmuir adsorption isotherm (A) of Fig.3 is related by gibbs energy by the following equation (Khamis 1990).

$$K_{ads} = \frac{1}{C_{H_2O}} \exp(-\Delta G/RT) \quad (10)$$

$$\text{It can be written as: } \Delta G = -2.303 RT \log (K_{ads} \cdot C_{H_2O}) \quad (11)$$

Where C_{H_2O} is the concentration of water in (ml / L) at metal/solution interface, R is universal gas constant and T is absolute temperature. Values of K_{ads} were calculated by the intercept of Langmuir isotherm $\log (\theta / 1-\theta)$ vs $\log C_{inh}$. The values of ΔG_{ads} were tabulated in Table 4. Obtained values of Gibbs energy were plotted against temperature in accordance with the following basic equation (El-Awady *et al.*, 1992).

$$\Delta G_{ads} = \Delta H_{ads} - T \Delta S_{ads} \quad (12)$$

Intercept of graph between ΔG_{ads} vs T give value of ΔH_{ads} and by putting the value of intercept in equation 12 values of ΔS_{ads} were obtained. These values are listed in Table 4.

The negative values of ΔG_{ads} indicated spontaneous corrosion inhibition process (Abd-El-Rahim *et al.*, 2001; Tang *et al.*, 2006; Tang *et al.*, 2003). Table 4 shows increase (values becomes less negative) in Gibbs energy with temperature, indicating the exothermic process, at which adsorption was unfavourable with increase in reaction temperature as a result of inhibitor desorption from the steel surface (Noor 2007). The values of ΔH_{ads} and ΔS_{ads} also predict the mechanism of inhibitor adsorption. The negative values of ΔG_{ads} are between -11.55 KJ/mol to -13.76 KJ/mol indicating physisorption of inhibitor species on steel surface. Generally values of ΔG_{ads} up to -20 KJ/mol are consistent with electrostatic interaction (physisorption) while more negative values than -40 KJ/mol are due to charge sharing from inhibitor species to metal surface to form a co-ordinate type of bond (Umoren *et al.*, 2009). The values of ΔH_{ads} are come out to be negative, shows exothermic adsorption process of adsorption of inhibitor on steel surfaces (Obi-Egbedi *et al.*, 2012), and this is further proved by lower inhibition efficiencies in Table 1 at higher temperatures.

The exothermic process is attributed to either physical or chemical adsorption or mixture of both (Bentiss *et al.*, 2005), whereas an endothermic process corresponds to chemisorptions (Lebrini *et al.*, 2011). In exothermic process values of ΔH_{ads} predicts physisorption or chemisorption process. For physisorption values of ΔH_{ads} is lower than 40 KJ/mol while for chemisorption it is approaches to 100 KJ/mol (Zarrouk *et al.*, 2010). The value of ΔH_{ads} in table 4 indicates physisorption process. The negative value of ΔS_{ads} in Table 4 shows decrease in entropy. This is agrees with what expected, when the adsorption is an exothermic process, it must be accompanied by a decrease in the entropy energy change and vice versa (Thomas and Thomas, 1981)

Conclusion

1. Result showed that AECPL is good corrosion inhibitor for mild steel in 1M HCl solution.
2. Corrosion rates increases with increase in temperature and decreases with increase in inhibitor concentration.
3. Inhibition efficiencies increases at lower temperature suggest the physisorption process of inhibitor on mild steel surface.
4. Apparent activation energy increases with increase in inhibitor concentrations also suggests physisorption.
5. Enthalpy of activation comes out to be negative which show endothermic process.
6. The values of Gibbs free energies calculated were negative shows spontaneity of corrosion inhibition process of mild steel in 1 M HCl in AECPL.

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