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# Corrosion inhibition study of zinc in 0.1M HNO<sub>3</sub>by diphenylcarbazide and caffeine

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# ABSTRACT

The corrosion behavior of Zn in 0.1 M HNO<sub>3</sub> solution containing various concentration of caffeine and diphenylcarbaziderange  $(2x10^4 - 1x10^4)$ <sup>3</sup>) M was investigated. The corrosion rates were measured by using weight loss measurement and polarization curve, The results of polarization method obtained showed that the rate of corrosion of zinc increased with increasing temperature from 293K to 323K and the values of inhibition efficiency of caffeine and DPC decreased with increasing the temperature and increased with increasingcaffeine and DPC concentrations, the highest protection efficiency reached to68.4% by Caffeine and 69.5% by DPC at293K and at (1x10<sup>-3</sup>) M caffeine and DPC concentrations by electrochemical method. The coverage  $(\theta)$  of metal surface by caffeine and DPC werebeing obtained from the rate of corrosion in the presence and absence of caffeine and DPCin the acid solution. Changes in the free energy, enthalpy and entropy associated with caffeine and DPC adsorption have been determined. Apparent activation energies have been calculated for the corrosion process of zinc in acidic media from Arrhenius Plots, which reach to highest value (15.625 kJ.mol<sup>-1</sup> and 12.953 kJ.mol<sup>-1</sup>) by using 5 x 10<sup>-4</sup> M DPC and 2 x 10<sup>-4</sup> M Caffeine respectively. © 2015 Trade Science Inc. - INDIA

#### **INTRODUCTION**

Corrosion is the destructive attack of a metal by chemical or electrochemical reaction with its environment. Corrosion occurs due to the metals spontaneous need to revert to a more stable form as it is found in nature<sup>[1-2]</sup>. Usually, the corrosion process consists of a set of redox reactions, which are electrochemical in nature. Thus, the metal is oxidized to corrosion products at anodic sites and some species are reduced at cathodic sites<sup>[3]</sup>. Zinc is one of the

## KEYWORDS

Corrosion; Inhibition; Caffeine; DPC; Zinc; Acidic solution.

most vital non-ferrousmetals, having extensive use in metalliccoating<sup>[4]</sup>. The influence of organic compounds containing nitrogen on the corrosion of zinc in acidic solutions has been investigated by several authors<sup>[5-8]</sup>. Aromatic, aliphatic and heterocyclic amines have been extensively investigated as corrosion inhibitors<sup>[9]</sup>. These organic compounds can adsorb on the metal surface, block the active sites on the surface and thereby reduce the corrosion rate. This paper describes a study of the protectionefficiency of caffeine and DPC for zinc

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corrosion in 0.1M HNO<sub>3</sub>.

## **EXPERIMENTAL**

#### Materials and reagents

Zinc 99.99% sheet were used for electrochemical and gravimetric studies. The zinc samples were mechanically polished using different grades of emery paper, washed with distilled water, and dried at room temperature. Appropriate concentration of aggressive solutions used (0.1M HNO<sub>3</sub>) was prepared using distilled water. Caffeine99.99% was obtained from (SID) samara Drugs industry and DPC from BDH, The structure of caffeine and DPC has been shown in Figure 1 and 2.

#### Weight loss method

In the weight loss experiments, the zinc samples were mechanically abraded using a series of emery paper from 400 to 1200 grade. The specimens were then rinsed with distilled water, degreased with acetone, rinsed again with distilled water and finally dried before being weighted and immersed in 75 cm<sup>3</sup> beaker containing the corrosive solution and different concentration of inhibitors at room temperature. At the end of the tests, the specimens were taken out from the solution at regular intervals of time (5 -10 min), rinsed with distilled water, degreased with acetone, dried and weighed using an analytical bal-



Figure 1 : Structure of caffeine



Figure 2 : Structure of diphenylcarbazide

ance (Sartorius). The zinc specimens were rectangular in the form  $(2 \times 1.5 \times 0.06)$  cm.

#### Potentiostatic polarization measurements

The polarization can be carried by using a potentiostat (M lab Potentiostat / galvanostat 200 (2007) (Germany) which was obtained from Bank Elektronik -- Intelligent Controls GnbH. Three electrode are required: the working electrode (WE) (that is the metal), the reference electrode (the potential of the WE is measured relative to this potential), and counter or auxiliary electrode (that the majority of the current passes through). Potentiostatic scans are especially suitable for an electrochemical process controlled by activation polarization. Anodic and cathodic polarization of zinc was carried out under potentiostatic conditions in 0.1M HNO<sub>2</sub> in the absence as well as in the presence of different concentrations of inhibitor from  $(2x10^{-4} - 1x10^{-3})$  M at different temperature (293-323) K. The working electrode was immersed in the test solution during 5 minutes until a steady state open circuit potential (E<sub>ocp</sub>) was obtained. The potential of according metal (WE) is varied (polarized) from its equilibrium value  $(E_{corr})$  firstly in the negative and then in the positive direction and the current response to the applied potential is recorded. The voltage/current density data pairs produced from the polarization, WE can then be used to construct polarization diagram<sup>[10-11]</sup>.

#### **RESULT AND DISCUSSION**

#### Weight loss measurement

Mass loss data for zinc corrosion in 0.1M HNO<sub>3</sub> in absence and presence of inhibitors, at various concentrations was studied. Figures 3 and 4 show the variation of weight loss with time for the corrosion of Zn in 0.1M HNO<sub>3</sub> containing various concentrations of DPC and Caffeine at 293 K. The Figures reveal that the weight loss of zinc decreases with increasing concentration of DPC and Caffeine, indicating that the rate of zinc corrosion in 0.1MHNO<sub>3</sub> decreases with increasing concentration of DPC and Caffeine.

The inhibition efficiency was increased with increasing concentration of DPC and Caffeine, there-



Figure 3 : Variation of weight loss with time for the corrosion of zinc in 0.1M HNO<sub>3</sub> containing various concentrations of DPC

Figure 4 : Variation of weight loss with time for the corrosion of zinc in 0.1M HNO<sub>3</sub> containing various concentrations of caffeine

TABLE 1 : Corrosion rate, percentage efficiencies and coverage degree for the corrosion 0f zinc in 0.1M HNO<sub>3</sub> in absence and presence of different concentrations of DPC and caffeine from weight loss measurements at 293K

Inhibitor	Conc. / M	<b>CR / mg. cm<sup>-2</sup>. h<sup>-1</sup></b>	%I	θ
Blank	0	33.1	-	_
	2 x 10 <sup>-4</sup>	16.58	50	0.5
DDC	5 x 10 <sup>-4</sup>	14.58	60	0.6
DPC	8 x 10 <sup>-4</sup>	11.14	66.3	0.663
	1 x 10 <sup>-3</sup>	10.3	68.8	0.688
Caffeine	2 x 10 <sup>-4</sup>	15.04	54.5	0.545
	5 x 10 <sup>-4</sup>	13.02	60.6	0.606
	8 x 10 <sup>-4</sup>	11.58	65	0.65
	1 x 10 <sup>-3</sup>	10.82	67.3	0.673

fore DPC and Caffeine act as adsorption inhibitors for the corrosion of zinc in HNO<sub>3</sub> solution<sup>[12]</sup>. This result suggests that the increase in efficiencies with increase in inhibitors concentration because of increases the number of molecules adsorbed onto Zinc surface and reduces the surface area that is available for the direct acid attack on the metal surface<sup>[13]</sup>. It was observed that DPC and Caffeine inhibit the corrosion of zinc in 0.1M HNO<sub>3</sub> solution, at all concentrations used in study, i.e. from  $2x10^{-4}$  to  $1x10^{-3}$ M. Maximum inhibition efficiency was obtained with 1x10<sup>-3</sup> M concentration of the inhibitor in 0.1M HNO<sub>2</sub> at 293K and reach to 68.8% by DPC and 67.3% by Caffeine. The corrosion rates of zinc in 0.1M HNO3 at 293K and the inhibition efficiencies by various concentrations of DPC and Caffeine are presented in TABLE 1. The results obtained from weight loss are in good agreement with electrochemi-

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cal studies. The corrosion rates of the zinc coupons have been determined for 1h immersion period at 293K from mass loss, using Eq. (1) where  $\Delta m$  is the mass loss, S is the area and t is the immersion period<sup>[14]</sup>. The percentage protection efficiency (%I) and coverage degree was calculated according the relationships Eq. (2) and (3) respectively<sup>[15]</sup>.

$$W = \Delta m / S. \Delta t \tag{1}$$

% I=
$$\frac{WB - Wi}{WB} \times 100$$
 (2)

$$\boldsymbol{\theta} = 1 - \frac{W_1}{W_2} \tag{3}$$

Where  $W_{\rm B}$  and  $W_{\rm i}$  are the Weight loss data of the metal coupons in the absence and presence of the inhibitor respectively. $\theta$  is the surface coverage.

#### Potentiostatic polarization measurements

The polarization curves of zinc recorded in 0.1 M HNO<sub>3</sub> in the absence and presence of DPC and

34

-600.00

-700.00

-800.00

-900.00

-1000.00

-1100.00

0.01

E (mV)

r r r r nul

BLANK +

0.0005 M 🔷

0.001 M



10.00



Figure 5 : Polarization plots of zinc in 0.1M HNO<sub>3</sub> for various concentrations of DPC at 313K

1.00 logl (mA/cm2)

0.10

Figure 6 : Polarization plots of zinc in 0.1M HNO<sub>3</sub> for various concentrations of Caffeine at 313K

TABLE 2 : Corrosion data of zinc in 0.1M HNO<sub>3</sub> at absence and presence different DPC concentrations at temperature ranges 293-323K

TTT

100.00

DPC M	T\K	-E <sub>corr</sub> mV	i <sub>corr</sub> μA/cm²	-b <sub>c</sub> mV/Dec	b <sub>a</sub> mV/Dec	θ	%I	Weight loss g/m².d	Penetration loss[mm/a]
	293	827.9	1670	137.6	136.8	_	-	489	25
0	303	837.1	1760	180.6	177.2	-	-	516	26.4
Blank	313	838.6	1920	173.0	176.2	-	-	563	28.8
	323	843.7	1990	158.5	168.7	-	-	581	29.7
	293	836.2	556.86	111.2	94.6	0.666	66.6	163	8.34
2 x 10 <sup>-4</sup>	303	842.3	662.11	113.5	78.7	0.623	62.3	194	9.92
2 X 10	313	841.7	745.44	97.9	99.6	0.611	61.1	218	11.2
	323	841.9	1040	163.0	153.3	0.477	47.7	305	15.6
	293	836.1	543.02	102.1	105.0	0.674	67.4	159	8.13
5 x 10 <sup>-4</sup>	303	837.1	645.41	112.0	98.8	0.633	63.3	189	9.67
5 X 10	313	843.7	738.30	107.6	119.1	0.615	61.5	216	11.1
	323	844.2	1010	121.6	147.2	0.492	49.2	295	15.1
	293	862.6	512.31	126.1	127.5	0.693	69.3	150	7.67
8 x 10 <sup>-4</sup>	303	869.3	625.88	96.3	109.0	0.644	64.4	183	9.37
8 X 10	313	869.6	724.11	115.1	108.7	0.622	62.2	212	10.8
	323	870.4	891.07	154.2	158.2	0.552	55.2	261	13.3
	293	822.4	509.10	95.8	84.9	0.695	69.5	149	7.63
$1 \times 10^{-3}$	303	824.2	618.29	109.5	99.6	0.648	64.8	181	9.26
1 x 10 <sup>-3</sup>	313	833.9	700.12	99.3	103.3	0.635	63.5	205	10.5
	323	840.5	860.53	171.3	175.6	0.567	56.7	252	12.9

Caffeine at different concentration at (293-323) K are presented in Figure 5 and 6. The extrapolation method for the polarizations curves was applied and the data for corrosion potential ( $E_{corr}$ ), corrosion current density ( $i_{corr}$ ), cathodic and anodic Tafel slopes ( $b_c$  and  $b_a$ ) and percentage inhibition efficiency (%I)

are shown in TABLE 2 and 3. In almost all these cases, the %I from Tafel plots agree well with the values obtained from weight loss data. In the case of polarization method the relation which used todetermine the inhibition efficiency (%I) was eq.4<sup>[16]</sup>:

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TABLE 3 : Corrosion data of zinc in 0.1M HNO<sub>3</sub> in absence and presence of different caffeine concentrations at temperature ranges 293-323K

Caffeine M	T\k	-E <sub>corr</sub> mV	i <sub>corr</sub> μA/ cm²	-b <sub>c</sub> mV/ Dec	b <sub>a</sub> mV/ Dec	θ	%I	Weight loss g/m².d	Penetration loss mm/a
	293	827.9	1670	137.6	136.8	-	-	489	25
	303	837.1	1760	180.6	177.2	-	-	516	26.4
0 Blank	313	838.6	1920	173.0	176.2	-	-	563	28.8
	323	843.7	1990	158.5	168.7	-	-	581	29.7
	293	848.5	553.34	113.0	131.1	0.668	66.8	162	8.29
2 x 10 <sup>-4</sup>	303	851.6	668.34	125.9	141.2	0.62	62	196	10
2 X 10	313	853.5	751.08	127.9	144.7	0.608	60.8	220	11.3
	323	855.3	939.17	93.4	106.3	0.528	52.8	275	14.1
	293	831.8	536.38	80.4	77.9	0.678	67.8	157	8.03
5 x 10 <sup>-4</sup>	303	842.2	645.80	130.7	104.4	0.633	63.3	189	9.67
J X 10	313	843.7	731.21	124.9	109.0	0.619	61.9	214	11
	323	845.7	875.60	126.2	128.0	0.56	56	256	13.1
	293	823.7	535.39	99.9	98.8	0.679	67.9	157	8.02
8 x 10 <sup>-4</sup>	303	843.9	625.89	129.4	128.0	0.644	64.4	183	9.38
8 X 10	313	845.2	713.12	113.8	101.7	0.628	62.8	209	10.7
	323	848.7	864.43	149.0	136.5	0.565	56.5	253	12.9
	293	832.8	527.16	81.0	77.8	0.684	68.4	154	7.90
$1 - 10^{-3}$	303	840.4	618.16	112.1	119.3	0.648	64.8	181	9.26
1 x 10 <sup>-3</sup>	313	847.0	707.27	108.3	125.5	0.631	63.1	207	10.6
	323	849.4	850.31	122.7	129.7	0.572	57.2	249	12.7

% I =  $(1 - i_{corr un} / i_{corri})$  100

Where  $i_{corr un}$  and  $i_{corri}$  are the uninhibited and inhibited corrosion current densities, respectively, determined by extrapolation of catholic Tafel lines to corrosion potential.

The tafel plots Figure 5 and 6 revel that the Corrosion potential values almost remained constant which indicate that inhibitors acts as mixed type of inhibitors<sup>[17]</sup>.

# **Kinetic of corrosion**

As noticed previously, the adsorption process was well elucidated by using a thermodynamic model; in addition a kinetic-thermodynamic model was another tool to explain the mechanism of corrosion inhibition for inhibitors. The apparent effective activation energies ( $E_a$ ) for the corrosion reaction of Zn in 0.1M HNO<sub>3</sub> in the absence and presence of different concentrations of investigated compounds were calculated from Arrhenius-type equation (5 and 6). A plot of log i<sub>corr</sub>(corrosion rate) vs. 1/T gave straight lines as shown in Figure 7 and 8.

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Inspection of TABLE 4 showed that the value of E determined in 0.1M HNO<sub>3</sub> containing DPC and Caffeine is higher than that for uninhibited solution. The increase in the apparent activation energy may be interpreted as physical adsorption that occurs in the first stage<sup>[18]</sup>. Szauer and Brand explained the increase in activation energy due to an appreciable decrease in the adsorption of the inhibitor on the zinc surface with increase in temperature. As adsorption decreases more desorption of inhibitor molecules occurs because these two opposite processes are in equilibrium. Due to more desorption of inhibitor molecules at higher temperatures the greater surface area of zinc comes in contact with aggressive environment, resulting increased corrosion rates with increase in temperature and decrease efficiency of inhibitors<sup>[19]</sup>.

# $CR = A \exp^{(-Ea/RT)}$

# $\log CR = \log A - E_a/2.303RT$

Where  $i_{corr}$  is the corrosion rate, R the gas constant, T the absolute temperature, A the pre exponential factor,  $E_a$  the activation energy for corrosion pro-

(5)

(6)



Figure 7 : Arrhenius plot of log  $i_{corr}$  versus 1/T for the corrosion of zinc in 0.1 M HNO<sub>3</sub> containing various DPC concentrations

Figure 8 : Arrhenius Plot of log  $i_{corr}$  versus 1/T for the corrosion of zinc in 0.1 M HNO<sub>3</sub> containing various caffeine concentrations

TABLE 4 : Activation energy ( $E_a$ ), pre exponential factor (A) and the entropy of activation ( $\Delta S^*$ ) for the corrosion of zinc in 0.1 M HNO<sub>3</sub> solution in the absence and presence of different concentration of DPC and caffeine over the temperature range 293 -323 K

Conc.				DPC	Caffeine				
/M 1	T/K	$\mathbf{R}^2$	E <sub>a</sub> kJ /mol	A/Molecule.cm <sup>-</sup> <sup>2</sup> . s <sup>-1</sup>	<b>∆</b> S <sup>*</sup> J.K <sup>-</sup> <sup>1</sup> .mol <sup>-1</sup>	R <sup>2</sup>	E <sub>a</sub> kJ /mol	A/Molecule.cm <sup>-</sup> <sup>2</sup> . s <sup>-1</sup>	∆ S <sup>*</sup> J.K <sup>-</sup> <sup>1.</sup> mol <sup>-1</sup>
	293				251.457				251.457
0	303	0.980	4.865	8.272 x10 <sup>25</sup>	251.17	0.980	4.865	8.272 x10 <sup>25</sup>	251.17
0	313				250.902				250.902
	323				250.634				250.634
	293				263.298				259.542
2x10 <sup>-4</sup>	303	0.932	15.187	3.431 x10 <sup>26</sup>	263.011	0.956	12.953	2.184 x10 <sup>26</sup>	259.255
2X10	313	0.932			262.743				258.987
	323				262.475				258.719
	293				262.585				258.768
5x10 <sup>-4</sup>	303	0.955	55 15.625	3.149 x10 <sup>26</sup>	262.298	0.994	12.545	1.99 x10 <sup>26</sup>	258.481
3X10	313	0.933			262.03				258.213
	323				261.761				257.945
	293				261.059				258.375
8x10 <sup>-4</sup>	303	0.994	94 14.211	2.621 x10 <sup>26</sup>	260.771	0.990	12.313	1.898 x10 <sup>26</sup>	258.088
8X10	313	0.994			260.503				257.82
	323				260.235				257.551
	293				259.767				258.344
1x10 <sup>-3</sup>	303	0.861	9.343	2.244 x10 <sup>26</sup>	259.48	0.993	12.323	1.891 x10 <sup>26</sup>	258.057
1X10	313				259.212				257.789
	323				258.944				257.521

cess, values of  $E_a$  and A then derived from the slope respectively. and the intercept of the plot of log  $i_{corr}$  versus 1/T Entropy of

Entropy of activation ( $\Delta S^*$ ) was then calculated



Figure 9 : Langmuir isotherm plot for the adsorption of DPC on the surface of zinc

from the value of A using the following relationship<sup>[20]</sup>:

$$\mathbf{A} = \mathbf{kT} / \mathbf{h} \, \mathbf{e}^{\,\Delta S^*/R} \tag{7}$$

$$Log A = kT / h + \Delta S^* / 2.303R$$
 (8)

When the activated complex represents a more probable arrangement of molecules than found in the normal reactant,  $\Delta S^*$  is positive and the reaction rate will be greater than normal. Conversely, when the activated complex results only after considerable rearrangement of the structure of the reactant molecules, making the complex a less probable structure,  $\Delta S^*$  is negative, and the reaction will be slower<sup>[20]</sup>.

#### **Adsorption isotherm**

The adsorption isotherm can be determined by assuming that inhibition effect is due mainly to the adsorption at metal solution interface. Basic information on the adsorption of inhibitors on the metal surface can be provided by adsorption isotherm. In order to obtain the isotherm, the fractional surface coverage values ( $\theta$ ) as a function of inhibitor concentration must be obtained. The values of  $\theta$  can be easily determined from the weight loss and electrochemical measurements by the ratio %I/100, where %I is inhibition efficiency obtained by weight loss and electrochemical method. So it is necessary to determine empirically which isotherm fits best to

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Figure 10 : Langmuir isotherm plot for the adsorption of Caffeine on the surface of zinc

the adsorption of inhibitors on the zinc surface. Several adsorption isotherms (viz., Frumkin, Langmuir, Temkin, Freundlich) were tested and the Langmuir adsorption isotherm was found to provide the best description of the adsorption behavior of these inhibitors. The Langmuir isotherm is given by following equation<sup>[21]</sup>:

$$\theta/1-=K_{ads}C$$
(9)

The rearrangement gives the following equation:

$$C/\theta = 1/K_{ads} + C$$
(10)

Where C (M) is the concentration of inhibitor,  $K_{ads}$  is the equilibrium constant of the adsorption process, and  $\theta$  is the surface coverage. Plot C/ $\theta$  versus C yields a straight line (Figure 9 and 10) with regression coefficient, R<sup>2</sup>, almost equal to 1. This suggests that DPC and caffeine in present study obeyed the Langmuir isotherm and there is negligible interaction between the adsorbed molecules. Free energy of adsorption was calculated using the relation<sup>[22]</sup>:

$$\mathbf{K}_{\mathrm{ads}} = 1/55.55 \, \exp^{(-\Delta \mathrm{Gads/RT})} \tag{11}$$

Where R is the universal gas constant and T is the absolute temperature. The value 55.55 in the above equation is the concentration of water in solution in mol L<sup>-1</sup>. The values of  $K_{ads}$  and  $\Delta G_{ads}$  were calculated at temperature range (293-223) K and are listed in TABLE 5.

Thermodynamic parameters were obtained from

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Inhibitor	T / K	$\mathbf{R}^2$	$K_{ads}$ / $M^{-1}$	-ΔG <sub>ads</sub> kJ mol <sup>-1</sup>	$-\Delta S_{ads} J mol^{-1}$	- <b>ΔH<sub>ads</sub> kJ mol<sup>-1</sup></b>
DPC	293	0.9998	50000	36.147		38.784
	303	1	33333	36.359		39.086
	313	0.9995	50000	38.614	9.00	41.431
	323	0.9951	10000	35.525		38.432
	293	1	111111	38.092		40.729
Caffeine	303	0.9999	50000	37.394		40.121
	313	1	50000	38.614	9.00	41.431
	323	0.9999	25000	37.986		40.893

TABLE 5 : Thermodynamic parameters for adsorption of the inhibitors on the surface of zinc in 0.1 M HNO<sub>3</sub>

Gibbs-Helmholtz equation according to this equation<sup>[23]</sup>:

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

Plots of  $\Delta G_{ads}$  vs. T for adsorption of the used compounds on the surface of zinc in 0.1M nitric acid over the temperature range from 293K to 323K. The data gave straight lines of slope  $\Delta S_{ads}$ . Also the standard adsorption entropy  $\Delta S_{ads}$  can be obtained, actually calculated on the basis of the equation (12):

 $-\Delta S_{ads} = (\partial \Delta G_{ads} / \partial T)_{p} (13)$ 

The value of  $\Delta H_{ads} \& \Delta S_{ads}$  were calculated and are listed in TABLE 5. The negative values of  $\Delta H_{ads}$  and  $\Delta S_{ads}$  obtained here indicate that the adsorption process is exothermic with an ordered phenomenon<sup>[24]</sup>.

The standard free energy of adsorption ( $\Delta G_{ads}$ ), calculated from Eq. (11). The negative value of the standard free energy of adsorption and the high values of the adsorption constant indicate a spontaneous adsorption of this inhibitor on zinc. This means that the inhibitive action of this substance result from the physical adsorption of these molecules on the surface of zinc<sup>[25-26]</sup>. This is also supported by the fact that the inhibition efficiency of the investigated inhibitors decreases at higher temperature (323K).

# CONCLUSION

- 1. The performance of these inhibitors can be optimized by taking advantages of the operating temperature and concentration of the inhibitors.
- 2. DPC and caffeineacts as good inhibitors for the corrosion of zinc in  $0.1M \text{ HNO}_3$ .
- 3. Potentiostatic curves reveal that DPC and caffeineis an anodic type of inhibitors.

- 4. The results obtained from weight loss and polarization studies are in good agreement with each other.
- 5. The adsorption of DPC and caffeineon zinc surface obeyed Langmuir adsorption isotherm.

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