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Research Article

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Adsorption and Inhibitive Properties of Januvia for the Corrosion of Zn in 0.1 M HCl

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Abstract The inhibitive and adsorptive characteristics of Januviafor the corrosion of zinc in HCl solutions have been studied using weight loss and gasometric methods of monitoring corrosion. The results obtained reveal that various concentrations of Januvia inhibited the corrosion of zinc in HCl between the temperature ranges of 303-333K. The inhibition efficiency of the inhibitor was found to decrease with temperature.Kinetic consideration reveals that the inhibited and uninhibited corrosion of zinc in the acid proceeded via first order kinetics with half-life values for the inhibited reactions being higher than values for the uninhibited systems. Thermodynamics consideration also reveals that this inhibitor is adsorbed on zinc surface by spontaneous physical adsorption mechanism. Values of free energy were lower than the threshold value of 40 kJ/mol. Also Temkin adsorption isotherms best described the adsorption characteristics of the inhibitor.

Keywords acid corrosion, weight loss, drugs, zinc, HCl

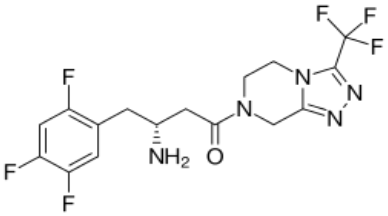
Introduction

Almost all metals are prone to corrosion and the level of corrosion attack depends on the type of metal and the condition of the environment. Several measures have been adopted to protect metals against corrosion including cathodic/anodic protection, greasing and galvanizing. However, the use of inhibitors has proved to be one of the best options. Corrosion inhibitors are substances, which will slow down the rate of corrosion when added in minute quantities [1]. Due to the toxicity of widely used corrosion inhibitors and the ever tightening environmental regulations surrounding their use and disposal, there is great interest in replacing harmful inhibitors with effective non-hazardous alternatives. Drugs (chemical medicines) seem to be ideal candidates to replace traditional toxic corrosion inhibitors because of their natural origin, as well as their non-toxic characteristics and negligible negative impacts on the aquatic environment [2-5], Several studies have been carried out and reported for the use of drugs as corrosion inhibitors. For example Cefuroxime axetil (CA) a pro-drug was tested as corrosion inhibitor for aluminum in hydrochloric acid solution by Ameh and Sani [6] using thermometric, gasometric weight loss and scanning electron microscope (SEM) techniques. Results obtained showed that this compound has good inhibiting properties for aluminum corrosion in acidic medium, with inhibition efficiencies values reaching 89.87 % at 0.5 g / L. The adsorption of the inhibitor was also found to be spontaneous, exothermic and best fitted the Langmuir adsorption model. Shukla and Quraishi [7] studied the inhibition efficiency of cefalexin drug for the corrosion mild steel in 1 MH Clusing electrochemical and weight loss measurements. The physical adsorption of the protonated cefalexin molecules on mild steel was suggested due the electrostatic attractions.

The inhibiting effect of streptoquin and septazole towards the corrosion of the copper in 0.1M of HCl solution was also studied by Fouda and Gadow [8] using potentiodynamic polarization measurements, electrochemical

impedance spectroscopy (EIS) and Electrochemical frequency modulation technique (EFM). They found out that the drugs were adsorbed on copper surface follow Langmuir adsorption isotherm.

In the present study, the inhibitive properties of this compound are reported for the first time, using weight loss measurement and gasometrictechniques. Sitagliptin (marketed as the phosphate salt under the trade name Januvia) is an oral antidiabetic drug of the dipeptidyl peptidase-4 (DPP-4) inhibitor class. This enzyme-inhibiting drug is used either alone or in combination with other oral antihyperglycemic agents (such as metformin or a thiazolidinedione) for treatment of diabetes mellitus type 2 [9]. The benefit of this medicine is its fewer side effects (e.g., less hypoglycemia, less weight gain) in the control of blood glucose values. While safety is its advantage, efficacy is often challenged as it is often recommended to be combined with other agents such as metformin. There have been several postmarketing reports of pancreatitis (some fatal) in people treated with sitagliptin and other DPP-4 inhibitors [10].



Sitagliptin

Materials

The sheets of zinc, A72357 type used for this study were obtained from the Novara group Limited England. Each sheet was 0.4 mm in thickness and were mechanically pressed cut into 5x4 cm coupons. The coupons were degreased by washing in absolute ethanol, dried in acetone and stored in moisture free desiccators before use [11-12]. Analar grade reagents were used. These included concentrated hydrochloric acid, ethanol and silver nitrate.

Preparation of samples

The concentration of acid (HCl) used for the study were 0.1M (for weight loss method) and 2.5M (for gasometric methods) while the concentrations of the inhibitor (Januvia) were 0.1, 0.2, 0.3, 0.4 and 0.5 g (per liter of the acid solution). These were dissolved in 1liter solution containing 0.1 M and 2.5 M of HCl respectively.

Corrosion monitoring

Weight loss method

A previously weighed metal (zinc sheet) was completely immersed in 250 ml of the test solution (different concentrations of acid, inhibitor as described in section 2.1) in an open beaker. The beaker was inserted into a water bath maintained at a temperature of 303 K. Similar experiments were repeated at 313 K, 323 K and 333 K. In each case, the weight of the sample before immersion was measured using Scaltec high precision balance (Model SPB31). Prior to measurement, each coupon was washed in 5 % chromic acid solution (containing 1% silver nitrate) and rinsed in de ionized water.

The difference in weight for a period of 168 hours was taken as total weight loss. The inhibition efficiency (% I) for each inhibitor was calculated using the formula [13],

$$\%I = \left(1 - \frac{W_1}{W_2}\right) \times 100 \qquad 1$$

where W_1 and W_2 are the weight losses (g/dm^3) for zincin the presence and absence of inhibitor in HCl solution respectively. The degree of surface coverage θ is given by the equation 2:

$$\theta = (1 - \frac{W_1}{W_2}) \qquad 2$$

The corrosion rates for zinc corrosion in different concentrations of the acid and other media have been determined for 168h immersion period from weight loss using equation 3 [14]

$$CR = \frac{w_1 - w_2}{At} \qquad 3$$

where CR is the corrosion rate of zinc, A is the surface area of the zinc coupon in cm^2 and t is the period of immersion in hours.

Gasometry method

In the hydrogen evolution measurement, the test solution (different concentrations of inhibitor, acid or their mixtures) was poured into the reaction vessel. Upon the introduction of zinc, the flask was quickly corked and the rise in volume of the paraffin due to hydrogen evolution was noted after every minute until a steady volume was observed. From the results obtained, the corrosion inhibition efficiency was calculated using the following equation,

$$\%IE = \frac{V_b - V_t}{V_b} \times 100 \qquad 4$$

where V_b is the volume of hydrogen gas evolved by the blank and V_t is the volume of hydrogen gas evolved in the presence of the inhibitor, after time, t.

Results and Discussion

Weight loss results

The results obtained for the variation of weight loss of zinc with time during the inhibition of the corrosion of zinc in solutions of HCl by Junivia at 303, 313, 323 and 333 K are presented in Figures 1 to 4 respectively. From the plots, it can be seen that weight loss of zinc increases with increase in the period of time and with increase in temperature indicating that the rate of corrosion of zinc increases with time and with increase in temperature. Also it was found that the weight loss of zinc decreases with increase in the concentration of the inhibitor indicating that the januvia is an adsorption inhibitor. For an adsorption inhibitor, weight loss is expected to decrease with increase in concentration of the inhibitor [15].

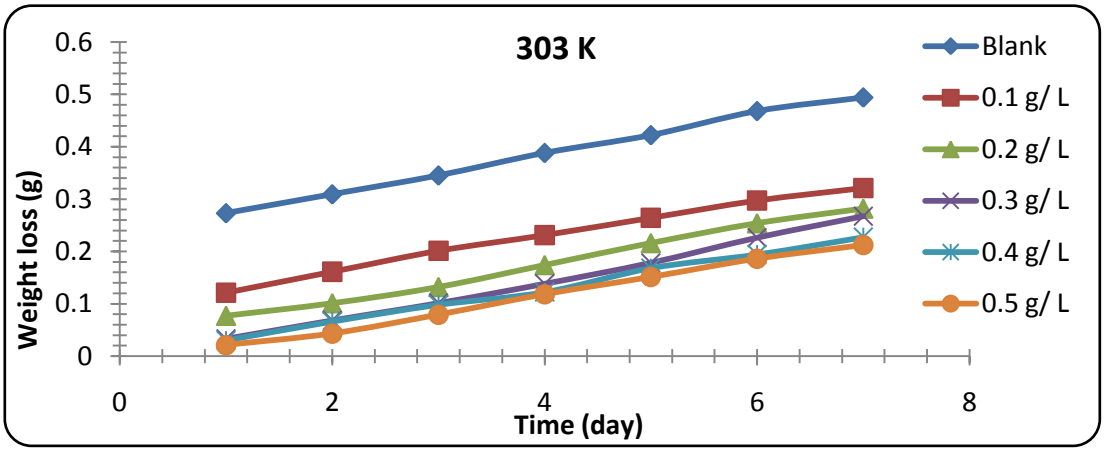


Figure 1: Variation of weight loss with time for the corrosion of zinc in 0.1 M HCl, containing various concentrations of Januvia as an inhibitor at 303 K

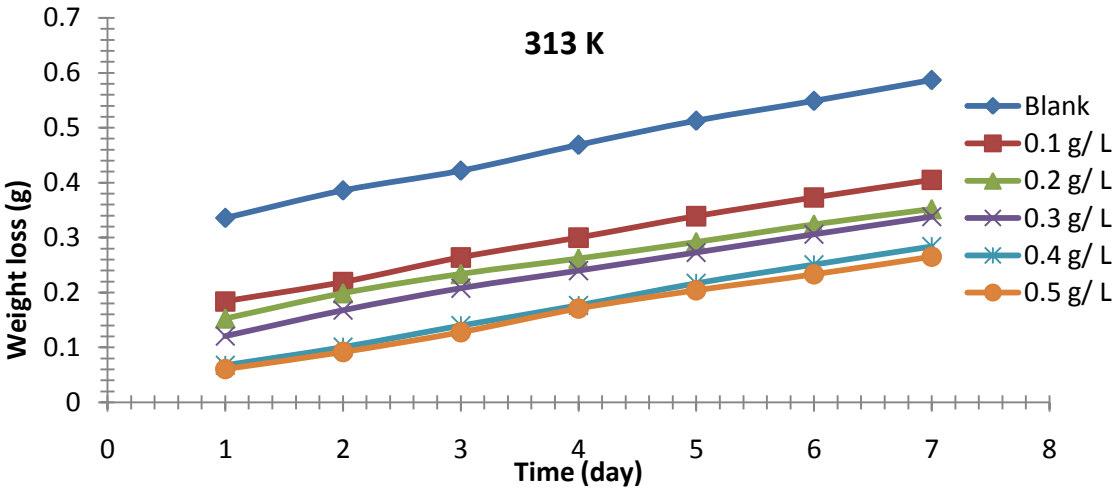


Figure 2: Variation of weight loss with time for the corrosion of zinc in 0.1 M HCl containing various concentrations of Januvia as an inhibitor at 313 K

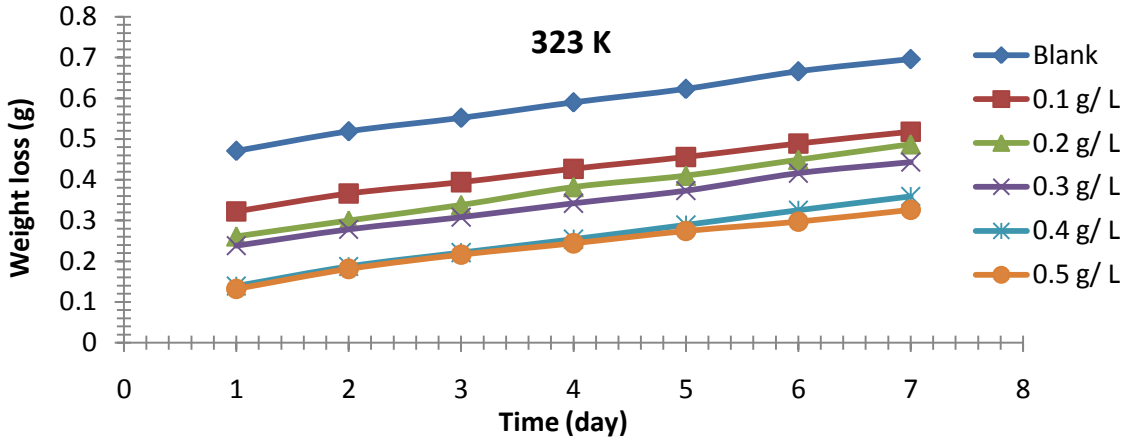


Figure 3: Variation of weight loss with time for the corrosion of zinc in 0.1 M HCl containing various concentrations of januvia as an inhibitor at 323 K

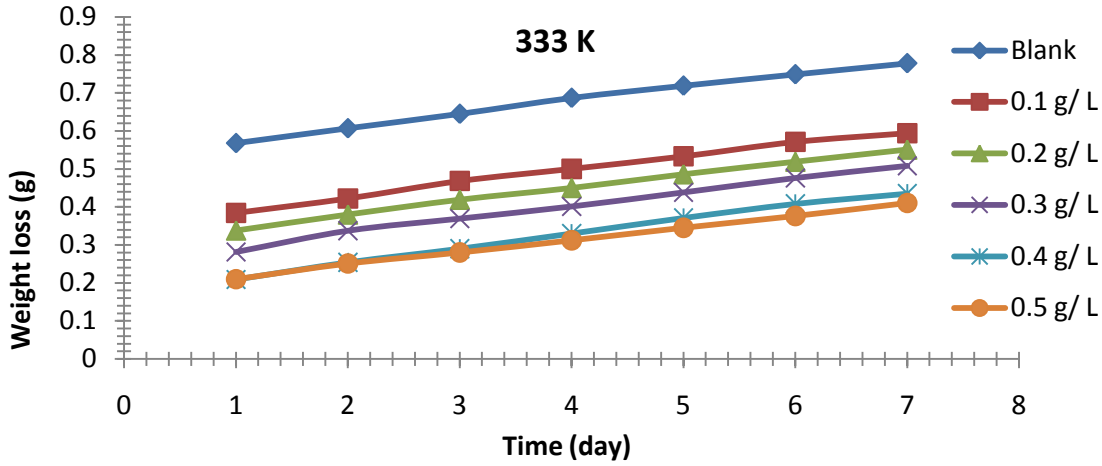


Figure 4: Variation of weight loss with time for the corrosion of zinc in 0.1 M HCl containing various concentrations of Januvia as an inhibitor at 333 K

Table 1: Corrosion rate of zinc 0.1 M HCl containing various concentrations of Januvia at 303, 313 323 and 333 K

	(CR)X10^{-5} (gcm^{-2} h^{-1})			
C (g/l)	303K	313K	323K	333K
Blank	15.30	19.80	24.20	26.60
0.1	13.60	16.40	22.10	24.10
0.2	11.80	13.70	19.20	21.80
0.3	10.40	12.10	16.50	18.30
0.4	8.80	11.10	13.80	15.70
0.5	8.30	10.10	11.70	14.00

The corrosion rates at different concentrations of Januvia at 303, 313, 323 and 333 K are summarized in the Table 1. From the table, it is clear that that the corrosion rates of zinc decreased with increase in the concentration of inhibitor. Values of inhibition efficiency and degree of surface coverage of Junivia calculated from equation 1 and 2 at 303, 313, 323 and 333 K are presented in Tables 2. As expected, inhibition efficiency of Januvia was found to increase with increase in concentration but decreases with increasing temperature. From the pattern of variation of inhibition efficiency with temperature, either of the two mechanisms of adsorption (physical and chemical adsorption) is feasible. However, the present results point toward physical adsorption as distinct from chemical adsorption, where the inhibition efficiency is expected to increase with increasing temperature [16]. Values of degree of surface coverage (θ) obtained, shows trend similar to those of inhibition efficiencies. This is because the degree of surface coverage is directly proportional to the inhibition efficiency.

The observed reversal of trend for corrosion rate and inhibition efficiency is an indication that the used inhibitor retarded the rate of corrosion of zinc in solution of HCl. Hence Januvia acted as an inhibitor for the corrosion of zinc in solution of HCl at temperatures between 303 to 333 K.

Gasometry method

Figure 5 shows the variation of the volume of hydrogen gas evolved with time for the corrosion of zinc in 2.5 M HCl containing various concentrations of Januvia at 303 K. From the figure, it was seen that the volume of hydrogen gas evolved increased with increase in the period of contact and with increase in temperature indicating that the rate of corrosion of Zinc in HCl (in the presence and absence of Januvia) increased with increase in temperature and increase in period of contact. The plots are also observed to display a pattern that is concentration dependent, in that the corrosion rate decreases with increase in the concentration of the inhibitor. Therefore, the studied inhibitor is an adsorption inhibitor. An adsorption inhibitor is characterized by decreasing rate of corrosion with concentration [15]. Values of inhibition efficiency, calculated from equation 4 are presented in Table 3. The values obtained are higher than those calculated from weight loss experiments indicating that the instantaneous inhibition efficiencies of Januvia is higher than their average inhibition efficiencies. Weight loss essentially measures inhibition efficiency over an average period of time while gasometric measures inhibition efficiency over a short period of time, usually, within an hour. Plots of variation of inhibition efficiency of Januvia obtained from weight loss with inhibition efficiency obtained from gasometric methods is given in Figure 6. From the plots it can be seen that results obtained from weight loss and gasometric methods were in excellent agreement ($R^2 = 0.976$).

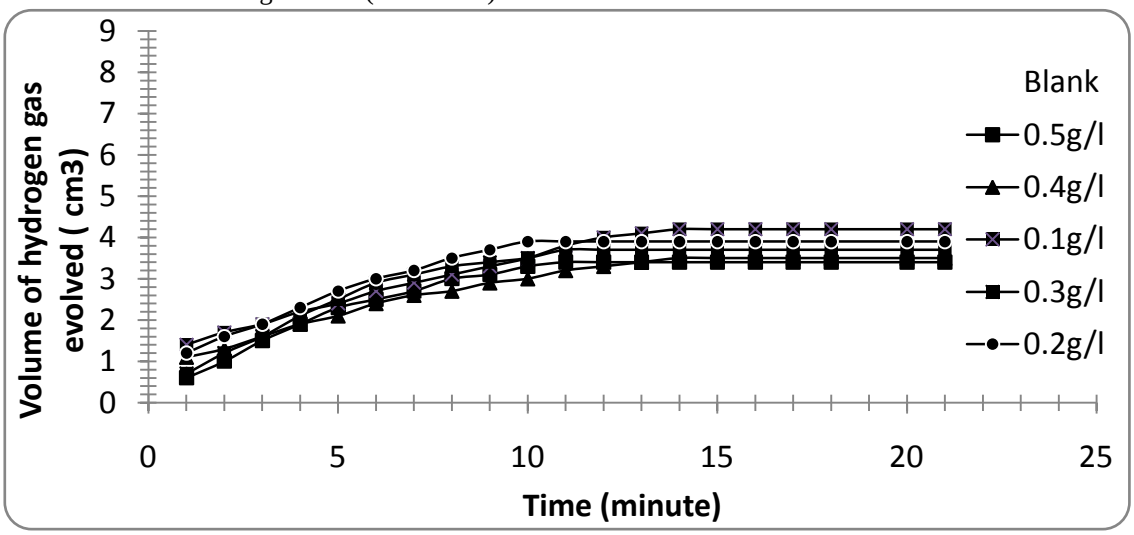


Figure 5: Variation of volume of hydrogen gas evolved with time for the inhibition of the corrosion of zinc in 0.1 M HCl by Januvia at 303 K

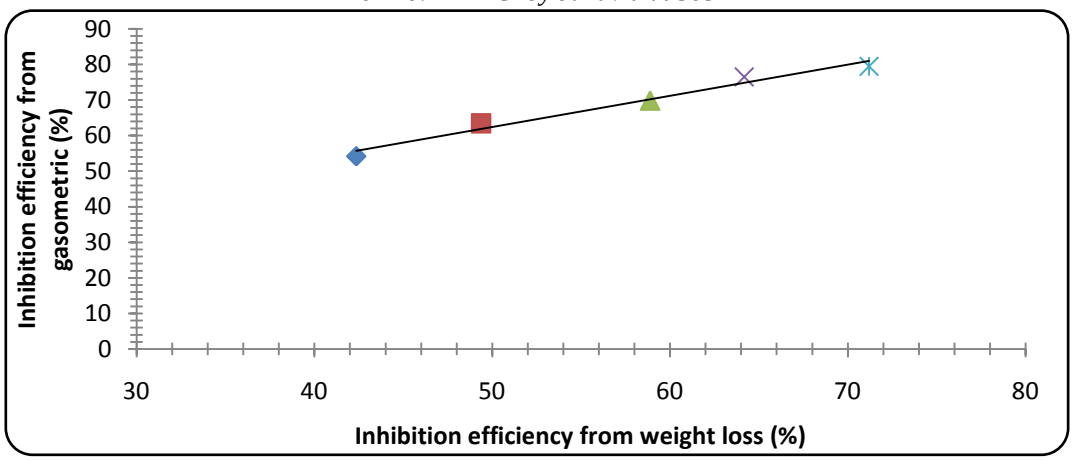


Figure 6: Variation of inhibition efficiency of Januvia obtained from weight loss with inhibition efficiency obtained from gasometric methods

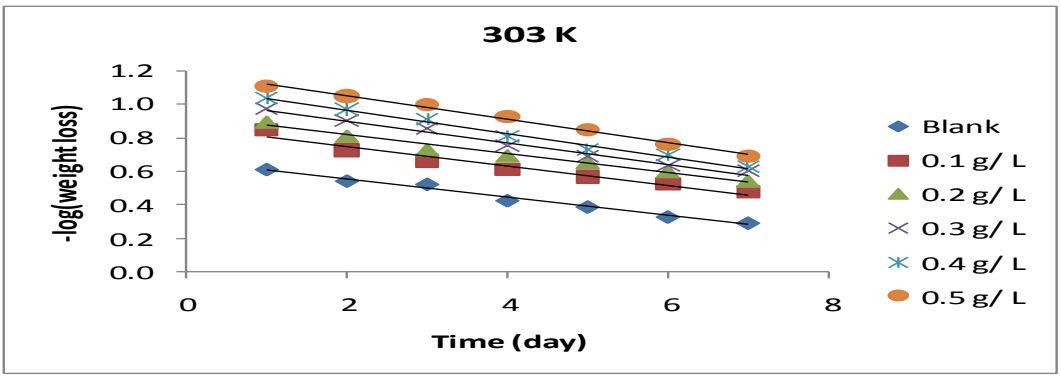


Figure 7: Variation of –log(weight loss) with time for the corrosion of zinc in 0.1 M HCl containing various concentrations of Januvia at 303 K

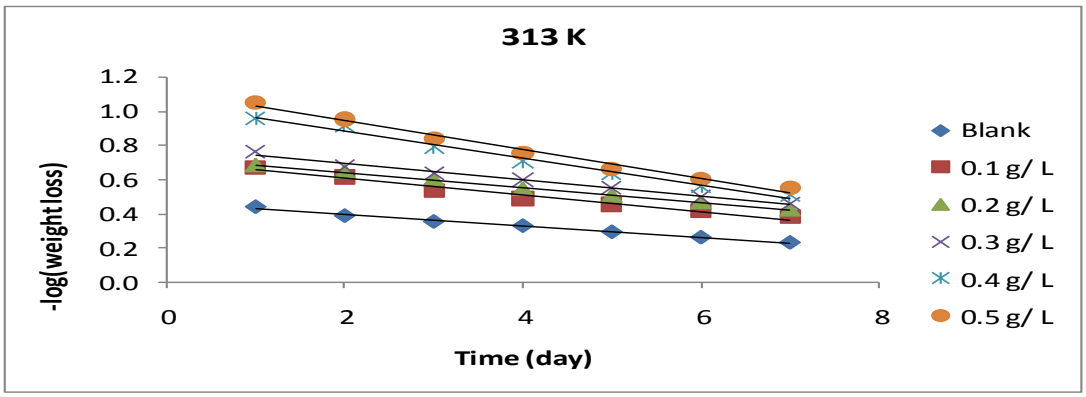


Figure 8: Variation of –log(weight loss) with time for the corrosion of zinc in 0.1 M HCl containing various concentrations of Januvia at 313 K

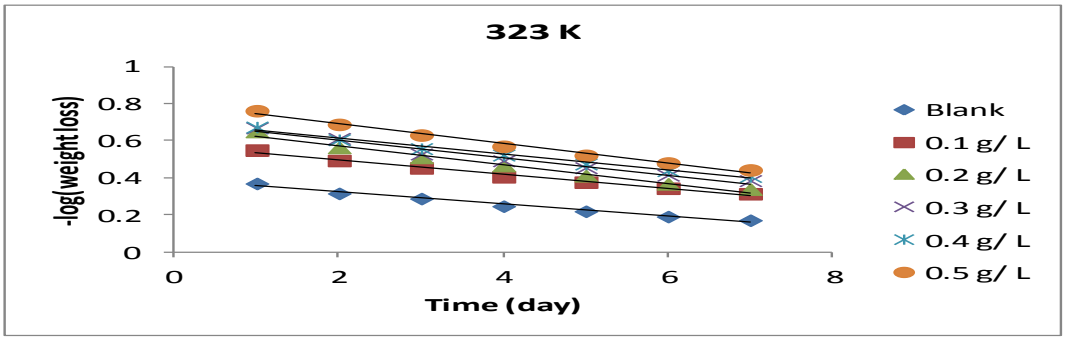


Figure 9: Variation of –log(weight loss) with time for the corrosion of zinc in 0.1 M HCl containing various concentrations of Januvia at 323 K

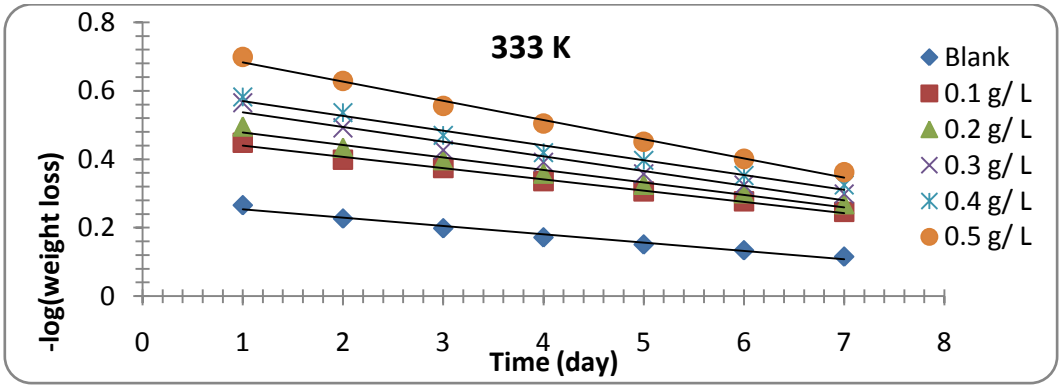


Figure 10: Variation of –log(weight loss) with time for the corrosion of zinc in 0.1 M HCl containing various concentrations of Januvia at 333 K

Kinetic study

The kinetic of the corrosion of zinc in HCl containing various concentrations of Januvia was studied by fitting weight loss data to different kinetic models. The test revealed that by plotting –log(weight loss) versus time, linear plots (Fig. 7 to 10) were obtained indicating that the corrosion of zinc in HCl is first order and that Equation 5 is applicable [16].

$$-\log(weight\ loss) = \frac{k_1 t}{2.303} \qquad \textbf{5}$$

where k_1 is the rate constant for the first order reaction and t is time (in days). A first order rate constant is related to the half-life ($t_{1/2}$) through, $t_{1/2} = 0.693/k_1$. From the value of k_1 calculated using Equation 5, values of half-life of the corrosion of zinc in the presence and absence of Januvia were computed and are recorded in Table 4. From the results obtained, it can be seen that the half-life of the inhibitor is greater than that of the blank indicating that the inhibitor increases the half-life of zinc in solution of HCl.

Table 2: Inhibition efficiency of Januvia for the corrosion of zinc in 0.1 M HCl at 303 to 333 K

	C (g/L)	303 K	313 K	323 K	333 K
Inhibition efficiency	**0.1**	42.35	37.28	32.42	29.44
	0.2	49.38	40.22	37.92	33.53
	0.3	58.89	47.28	44.10	39.83
	0.4	64.17	51.25	47.55	42.94
	0.5	71.20	56.02	55.62	47.42
Degree of surface coverage	**0.1**	0.4235	0.3728	0.3242	0.2944
	0.2	0.4938	0.4022	0.3792	0.3353
	0.3	0.5889	0.4728	0.4410	0.3983
	0.4	0.6417	0.5125	0.4755	0.4294
	0.5	0.7120	0.5602	0.5562	0.4742

Table 3: Inhibition efficiencies of Junivia calculated from hydrogen evolution measurement

C (g/L)	Inhibition efficiency
0.1	54.20
0.2	63.48
0.3	69.87
0.4	76.50
0.5	79.52

Table 4: Kinetic parameters for the inhibition of the corrosion of zinc in 0.1 M HCl by Januvia

	System	Slope	K_1	$t_{1/2}$ (day)	R^2
303 K	**Blank**	0.0702	0.1617	4.28	0.988
	0.1 g/L	0.0694	0.1598	4.33	0.972
	0.2 g/L	0.0644	0.1483	4.67	0.977
	0.3 g/L	0.0571	0.1315	5.26	0.989
	0.4 g/L	0.0551	0.127	5.45	0.993
	0.5 g/L	0.0531	0.1224	5.66	0.974
313 K	**Blank**	0.0850	0.1958	3.53	0.980
	0.1 g/L	0.0798	0.1838	3.77	0.995
	0.2 g/L	0.0485	0.1117	6.20	0.994
	0.3 g/L	0.0481	0.1108	6.25	0.988
	0.4 g/L	0.0439	0.1011	6.85	0.990
	0.5 g/L	0.0337	0.0776	8.93	0.988
323 K	**Blank**	0.0530	0.1221	5.67	0.987
	0.1 g/L	0.0433	0.0997	6.95	0.981
	0.2 g/L	0.0510	0.117	5.92	0.985
	0.3 g/L	0.0473	0.1089	6.36	0.979
	0.4 g/L	0.0387	0.0891	7.77	0.985
	0.5 g/L	0.0326	0.0751	9.22	0.986
333 K	**Blank**	0.0561	0.1292	5.36	0.982
	0.1 g/L	0.0432	0.0995	6.96	0.976
	0.2 g/L	0.0429	0.0988	7.01	0.985
	0.3 g/L	0.0364	0.0838	8.26	0.973
	0.4 g/L	0.0328	0.0755	9.17	0.975
	0.5 g/L	0.0244	0.0562	12.33	0.988

Table 5: Arrhenius parameters for the inhibition of the corrosion of zinc by Januvia in 0.1 M HCl

System	E_a (J/mol)	A	R^2
Blank	11.84	0.0000262	0.996
0.1 g/L	16.51	0.0000932	0.989
0.2 g/L	16.31	0.0000825	0.993
0.3 g/L	16.14	0.0000651	0.994
0.4 g/L	16.00	0.0000424	0.964
0.5 g/L	16.97	0.0000555	0.995

Table 6: Thermodynamic parameters for the adsorption of various concentration of Januvia on zinc surface in 0.1 M HCl

System	ΔH^o_{ads} (kJ mol^{-1})	ΔS^o_{ads} (J mol^{-1} K^{-1})	R^2
Blank	8.82	-159.75	0.923
0.1 g/L	12.75	-142.45	0.974
0.2 g/L	11.09	-138.20	0.9773
0.3 g/L	14.26	-133.33	0.9887
0.4 g/L	15.75	-129.24	0.9287
0.5 g/L	22.22	-126.06	0.9302

Table 7: Temkin parameters for the adsorption of Januvia on zinc surface at various temperatures in 0.1 M HCl

T (K)	a	ln b	ΔG^o_{ads} (kJ mol^{-1})	R^2
303	-3.34	0.1075	-10.39	0.984
313	-3.63	0.0850	-10.67	0.968
323	-4.30	0.0631	-10.96	0.965
333	-4.88	0.0515	-11.26	0.949

Effect of temperature

The effect of temperature on the corrosion of zinc in the absence and presence of various concentration of the inhibitor was investigated using the Arrhenius equation, which can be written as follows [17],

$$CR = \mathrm{A}\exp\left(\frac{E_a}{RT}\right) \qquad 6$$

From the logarithm of equation 6, equation 7 was obtained:

$$\ln(CR) = \ln A - \frac{E_a}{RT} \qquad 7$$

where CR is the corrosion rate of zinc, A is the Arrhenius or pre-exponential factor, R is the universal gas constant and T is the temperature. Equation 7 reveals that a plot of ln(CR) versus 1/T is expected to be linear, with slope and intercept equal to $-E_a/R$ and ln A respectively.

Figure 11 shows the Arrhenius plots for the corrosion of zinc in solution of HCl, in the absence and presence of various concentrations of the inhibitor. Parameters deduced from the plots are presented in Table 5. It is evident from the results obtained that the present data fitted the Arrhenius model with a high degree of linearity ($0.9910 \leq R^2 \geq 0.9960$). Also, the activation energies deduced from the slopes of the plots ranged from 11.84 to 16.97 J/mol. These values are lower than the threshold value of 80 kJ/mol required for the mechanism of chemical adsorption implying that the adsorption of the studied inhibitor supports the mechanism of physical adsorption. Interestingly, the activation energy for the blank was least but the values (E_a) increased with increase in the concentration of the inhibitor indicating that the inhibition process is strongly activated.

Thermodynamic/adsorption considerations

The mechanism of inhibition of the corrosion of zinc by Januvia is found to be physical adsorption indicating that the extent of adsorption decreases with increase in temperature. A critical insight into the nature and feasibility of adsorption can be gotten through thermodynamic/ adsorption parameters. Therefore, the standard enthalpy and entropy changes of adsorption of Januvia were estimated through the Transition state plots. The Transition state equation, relates the corrosion rate with these parameters according to equation 8 [18].

$$CR = \frac{RT}{Nh} exp\left(\frac{\Delta S^0_{ads}}{R}\right) exp\left(\frac{-\Delta H^0_{ads}}{RT}\right) \qquad 8$$

where N is the Avogadro' s number, h is the plank constant, ΔS^0_{ads} is the standard entropy change of adsorption, ΔH^0_{ads} is the enthalpy change of adsorption. From the logarithm and rearrangement of equation 8, equation 9 was obtained:

$$ln\left(\frac{CR}{T}\right) = ln\left(\frac{R}{Nh}\right) + \frac{\Delta S^0_{ads}}{R} - \frac{\Delta H^0_{ads}}{RT} \qquad 9$$

The expression in equation 9 is a linear model, indicating that a plot of $ln\left(\frac{CR}{T}\right)$ versus 1/T should give a straight line with slope and intercept equal to - $\frac{\Delta H^0_{ads}}{R}$ and $(ln\left(\frac{R}{Nh}\right) + \frac{\Delta S^0_{ads}}{R})$ respectively. The Transition state equation for the corrosion of zinc in solution of HCl in the absence and presence of various concentrations of Januvia is shown in Figure 12.

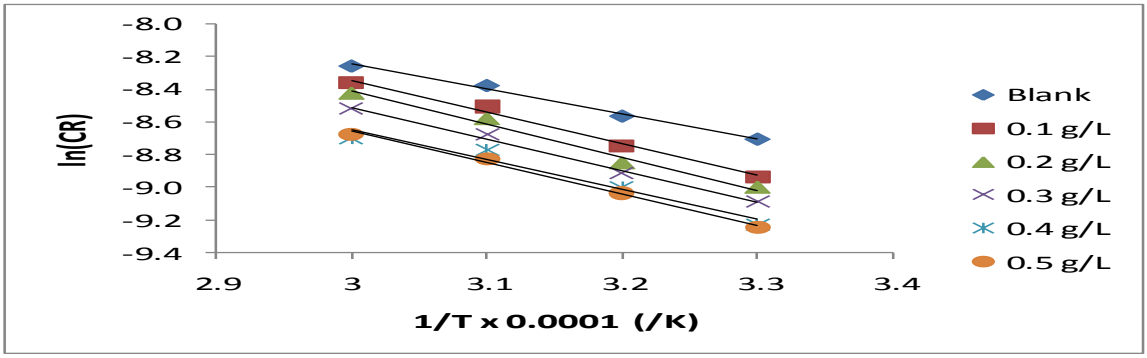


Figure 11: Arrhenius plots for the inhibition of the corrosion of zinc in solution of HCl by Januvia

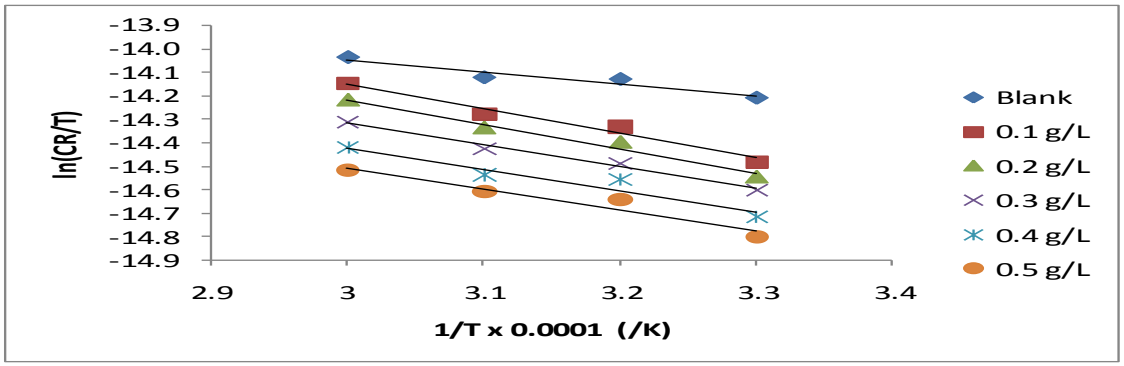


Figure 12: Transition state plots for the inhibition of the corrosion of zinc in solution of HCl by Januvia

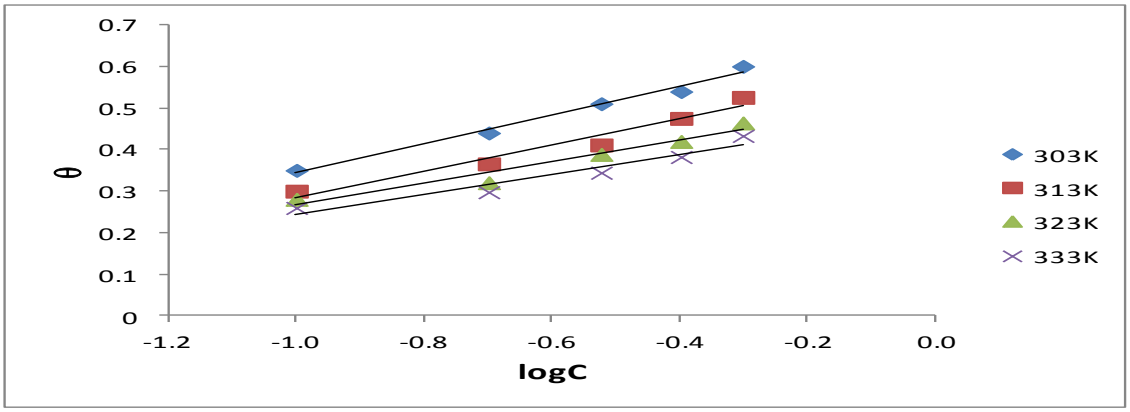


Figure 13: Temkin isotherm for the adsorption of Januvia on Zinc surface at various temperatures.

Adsorption parameters deduced from the plots are recorded in Tables 6. From the results obtained, high degrees of linearity were obtained for all the plots indicating that the adsorption of this inhibitor fitted the Transition state model. Values of enthalpy change of adsorption, calculated from the slope of the plots were found to be positive indicating that the adsorption of the inhibitor on the surface of zinc is endothermic. On the other hand, calculated values of entropy change of adsorption were negative indicating that there is an increase in randomness in moving from reactants to the activated complex during adsorption [19].

The inhibition of zinc corrosion in the presence of the inhibitor has been attributed to their adsorption onto the metal surface and this is usually confirmed from the fit of the experimental data to various adsorption isotherms. The adsorption process is influenced by the nature and surface charge of the metal, by the type of aggressive electrolyte and by the chemical structure of the inhibitors. The gravimetric data from the present study were fitted to the most frequently used adsorption isotherms, namely: Temkin, Frumkin, Langmuir, Freundlich, Hill de Boer, Parsons, Flory Huggins, Dhar-Flory Huggins, and BockrisSwinkles, and the correlation coefficients (R^2) were used to determine the best fits.

All of these isotherms are of the general form [20-21]

$$f(\theta, x)\exp(-2a\theta) = b_{ads}C \qquad 10$$

where $f(\theta, x)$is the configurational factor which depends upon the physical model and assumption underlying the derivative of the isotherm, $\theta$is the surface coverage, C is the inhibitor concentration, x is the size ration, 'a' is the molecular interaction parameter and b_{ads} is the equilibrium constant of adsorption process. From the fitness of the data to various adsorption isotherm, the Temkin adsorption isotherm, which is expressed according to equation 11 was found to be the best isotherm [22].

$$\exp(-2a\theta) = b_{ads}C \qquad 11$$

By rearranging and taking the logarithm of equation 11, equation 12 was obtained

$$\theta = \frac{-2.303}{2a} log b_{ads} - \frac{2.303}{2a} log\ C \qquad 12$$

The mathematical implication of equation 12 is that a plot of θ versus log C should be linear with slope and intercept equal to - $\frac{2.303}{2a}$ and - $\frac{2.303}{2a}$ log b_{ads} respectively. Figure 13 shows Temkin isotherm for the adsorption of Januvia on zinc surface respectively. Adsorption parameters deduced from the plots are presented in Tables7. The data reveal that high degrees of linearity were observed for all the inhibitors and that interaction parameters were positive, indicating the attractive behaviour of the inhibitors. It is significant to note that the equilibrium constant of adsorption obtained from the Temkin adsorption model (b_{ads}) is related to the standard free energy of adsorption according to equation 13[18]

$$lnb_{ads} = -\frac{1}{55.5}\ exp\left(\frac{\Delta G^0_{ads}}{RT}\right) \qquad 13$$

Values of ΔG^0_{ads} calculated from equation 13, are also presented in Tables 7. The free energies are werefound to range from -11.26 to -10.39 kJ / mol. Classically, free energy values less negative than – 40 kJ/mol is consistent with the mechanism of physical adsorption while free energy values more negative than -40 kJ/mol is consistent with the mechanism of chemical adsorption. (Ameh, 2015). Therefore the adsorption of Januvia on zinc surface is spontaneous and supports the mechanism of physical adsorption

Conclusion

From the results and findings of this study, it can be concluded that Januvia is a good adsorption inhibitor for zinc in acidic medium. The inhibitor acted through the mechanism of physical adsorption and their adsorption behaviour is best described by the Temkin adsorption models. Data obtained from gravimetric and gasometric are in good agreement with each other.

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