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Joint effect of halides and Ethanol Extract of Sorghum on the Inhibition of the Corrosion of Aluminum in HCl

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Abstract *In order to applied the principle of resource recovery to the management of sorghum waste from a brewery industry in Nigeria, ethanol extract of the waste was investigated for their corrosion inhibition efficiency towards aluminum in solution of HCl . Weight loss and gasometric methods were used for the study. The results obtained indicated that ethanol extract of sorghum waste inhibited the corrosion of aluminum in solution of HCl with maximum inhibition efficiency of 44.99, 39.95, 38.95 and 38.42% at 303, 313, 323 and 333 K respectively. However, through synergistic combination of KBr, KI and KCl, the inhibition efficiencies at 303 K was increase to 78.59, 54.22 and 79.04% respectively. Maximum efficiency obtained from gasometric experiment was 79.32%. The inhibitor acted as adsorption inhibitor and its adsorption behaviour obeyed Temkin, Freundlich, El awardy et al and Dubinin-Raduskevich isotherm. Data obtained with respect to trends of variation of inhibition efficiency with temperature, calculated activation and free energies as well as thermodynamic parameters confirmed that the adsorption of the inhibitor is spontaneous, exothermic and is consistent with the mechanism of charge transfer from charged inhibitor to charged metal surface, which favour physical adsorption.*

Key Words: *Corrosion, aluminum, inhibition, ethanol extract of sorghum waste, gravimetric and gasoetric investigation*

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1.0 Introduction

Current trend in the management of corrosion inhibitor involves the use of materials that are eco-friendly, less expensive, biodegradable in addition to better inhibition efficiency (Ngobiri *et* al., 2015). The science of waste management including waste recycling and recovery have been extended to the field of corrosion. In our research group, we have used mango peel waste, banana and plantain peels wastes, and orange peel waste for corrosion inhibition and good inhibition efficiencies were obtained (Eddy and Ebenso, 2008, Eddy *et al.,* 2008*;* Ukpe *et al*., 2014; Ukpe, 2019). Other have reported the use of aqueous extract of agro-industrial waste to inhibit the corrosion of stainless steel in sulphuric acid and inhibition efficiency up to 82% was obtained (Matosa *et al*., 2019). Ismail *et al*. (2011) reportedly used solid wastes for the inhibition of the corrosion of mild steel in acidic medium and observed that the wastes were good green corrosion inhibitors with demonstrated high inhibition efficiencies. Stango and Vijayalakshm (2018) have also reported maximum inhibition efficiency of 99.19% for orange peel used in the inhibition of mild steel corrosion.

The use of plant waste as corrosion inhibitor is based on their phytochemical constituents which presents structures that may be aromatic, rich in hetero atoms, possession of pi-electron and suitable functional groups. In continuation of researches on resource recovery and corrosion inhibition, the present study is aimed at investigating the corrosion inhibition potential of sorghum waste for aluminum corrosion in solution of HCl. The study is also supported by synergistic study in order to enhance the adsorption of the inhibitor on the metal surface.

2.0 Materials and Methods

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2. 1 Materials

The composition of the aluminum sheet was (wt %) Mn (0.6), P (0.36), C (0.15) and Si (0.03). Several metal coupons of dimension 5 x 4cm were produced from the sheet. The coupons were degreased by washing in absolute ethanol, dried in acetone and stored in moisture free desiccators before use (Agrawal *et al*., 2003; Jovancicervic *et al*., 1999; Ukpe *et al*., 2019). Analar grade reagents used for the study included concentrated hydrochloric acid,

ethanol and aluminum dust. Sorghum wastes (OPW) were obtained from Champion Brewery, Uyo, Akwa Ibom State, Nigeria. The waste materials were sun-dried, grounded into power sample and soaked in ethanol for one day. The soaked samples were respectively subjected to Soxhlet extraction and the ethanol extracts obtained for each sample were stored for used.

0.1 M HCl was prepared from stock solution of the acid and was stored in twenty-four different containers. The first container was labelled as blank. Stock solution containing 0.1, 0.2, 0.3, 0.4 and 0.5 g of a given sample (per liter of the acid solution) were respectively prepared and preserved for use.

2. 2 Weight loss measurements

Previously weighed metal (aluminium sheet) was completely immersed in 250 ml of the test solution (different concentrations of acid, halides, or inhibitors) 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, 323, and 333 K. In each case, the weight of the sample before immersion was measured using Scaltec high precision balance (Model SPB31) After every 24 hours, each sample was removed from the test solution, washed in a solution of NaOH containing aluminium dust and dried in acetone before re-weighing. The difference in weight after 168 hours of immersion was recorded as total weight loss and from the weight loss, the inhibitor was calculated using equation 1 (Eddy *et al*., 2011a),

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

where W_1 and W_2 are the weight loss (g) for aluminium in the presence and absence of the inhibitor. The degree of surface coverage θ is given by the equation 2

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

Also, the corrosion rates of the metal was calculated using equation 3

$$Corrosion\ rate\ (mpy) = \frac{534W}{DAT} \qquad (3)$$

where W = weight loss (mg); D = density of specimen (g/cm^3), A = area of specimen (square inches) and T = period of immersion (hour).

2.3 Synergistic study

Gravimetric method was also adopted for synergistic study but each concentration of the test solution was mixed with 0.06M of the respective halides (namely, KBr, KI and KCl), from the calculated inhibition efficiencies, synergistic parameters were calculated using the following equation (Eddy *et al*., 2013),

$$S = \frac{1 - \eta_A + \eta_A\eta_B}{1 - \eta_{AB}} \qquad (4)$$

2.4 Hydrogen evolution experiments

In gasometric experiemnt, the metal was inserted into a three-neck thermometric flask (which has provision for introducing thermometer, test solution and the metal). The system was properly lagged and changes in temperature were recorded every one minute. The reaction number and the inhibition efficiency were calculated using equations 4 and 5 respectively (Ameh and Eddy, 2018a).

$$RN = \frac{T_t - T_0}{T_t} \qquad (5)$$

$$\%\, I = \frac{\mathrm{RN}_{aq} - \mathrm{RN}_{wi}}{\mathrm{RN}_{aq}} \times \frac{100}{1} \qquad (6)$$

where V_b is the volume of hydrogen gas evolved by the blank and V_t is the volume of H_2 evolved in the presence of the inhibitor, after time, t.

3.0 Results and Discussion

3.1 Corrosion inhibition

Fig. 1 shows plots for the variation of weight loss with time during the corrosion of aluminum in solution of HCl containing various concentrations of ethanol extract of sorghum waste (SHW). The observed trends indicated that corrosion rate of aluminum increases with increase in the period of contact (of the metal with the solution) and with increase in temperature. However, the corrosion rate of aluminum was also observed to decrease with increase in the concentration of SHW, which indicated that SHW inhibited the corrosion of aluminum in solution of HCl and that the inhibition efficiency increases with increase in concentration of SHW. Therefore, SHW is an adsorption inhibitor for the corrosion of aluminum (Awe *et al*., 2019). This also implies that the inhibition efficiency of SHW for aluminum increases with concentration as shown in Table 1. The observed increase in corrosion rate with temperature (Table 1) indicates that the inhibition efficiency decreases with temperature which reflects the mechanism of physical adsorption (Odoemelam *et al*., 2009).

Although inhibition efficiencies obtained from gasometric measurements were higher than those obtained from weight loss experiments, both set of data correlated strongly with each other (R^2 = 0.976).

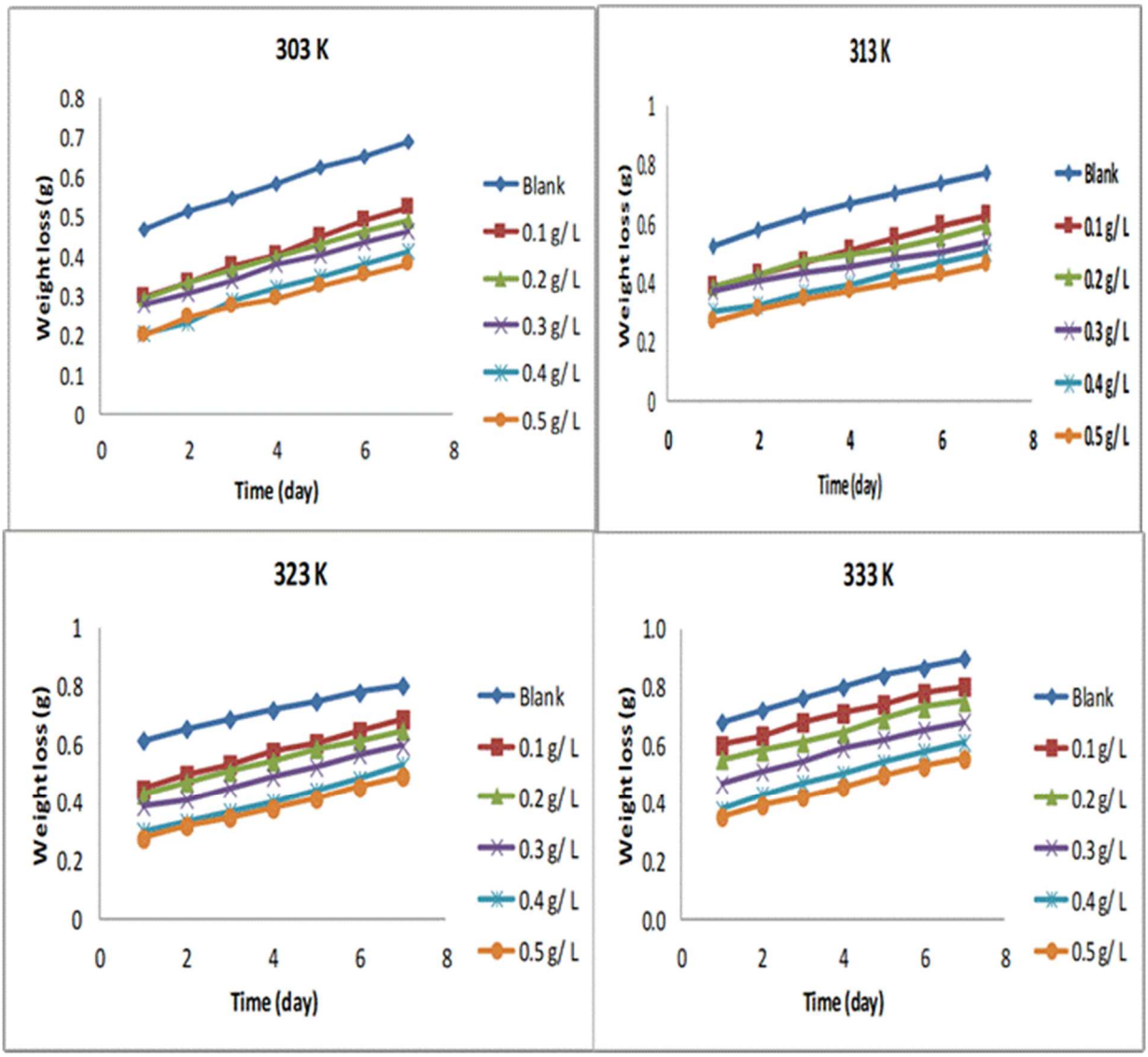

Fig. 1: Variation of weight loss with time for the corrosion of aluminium in 0.1 M HCl, containing various concentrations of sorghum (SGW) waste as additives at 303 to 333 K

Table 1: Corrosion rate (CR x 0.0001) of aluminium and inhibition efficiency of ethanol extract of SHW in solutions of HCl at 303 to 333 K

C (g/L)	Corrosion rate x 0.001 (mpy)				Inhibition efficiency (%)							
	303K	313K	323K	333K	303K	313K	323K	333K	KBr+ SHW	KI + SHW	KCl+ SHW	HE
Blank	2.99	3.44	3.65	3.77	-	-	-	-				
0.1	2.05	2.29	2.38	2.67	23.95	18.36	14.36	10.69	64.77	40.27	47.02	66.22
0.2	1.56	1.87	2.04	2.39	29.03	23.44	19.35	16.48	64.79	40.44	50..38	69.44
0.3	1.46	1.75	1.92	2.23	32.95	29.95	25.97	24.16	75.46	46.07	58.66	73.24
0.4	1.38	1.60	1.76	2.03	40.06	35.03	33.96	32.07	76.52	50.22	77.66	75.39
0.5	1.23	1.49	1.57	1.82	44.99	39.97	38.95	38.42	78.59	54.22	79.04	79.32

**** HE= Inhibition efficiency of SHW from hydrogen evolution experiment**

The difference is explained as a consequence of instantaneous inhibition efficiency been better than the average inhibition efficiency obtained from weight loss measurements.

In the presence of halides ions (0.06 M KCl, KI and KBr respectively), the inhibition efficiency was greatly enhanced (Table 1). In order to ascertain synergistic or antagonistic interaction, synergistic parameters were calculated using equation 4 and the results obtained are recorded in Table 2.

Table 2: Synergistic parameters (S) for SHW in combination with 0.06MKI, 0.06MKBr and 0.06MKCl respectively at 303K

Con of SHW. (g/dm3)	KBr + SHW	KI + SHW	KCl + SHW
0.1	4.71	0.27	7.02
0.2	4.72	0.04	21.38
0.3	5.96	0.00	8.70
0.4	6.92	0.15	7.66
0.5	8.56	0.02	7.04

The results backed the evidence that, although KI increase the inhibition efficiency of SHW for mild steel, calculated synergistic parameters (which were less than unity) indicated that there was no synergistic interaction between SHW and KI. However, synergistic parameters calculated for KBr and KCl were greater than unity, hence KBr and KCl synergistically enhance the adsorption of SHW on aluminum surface.

3. 2 Kinetic study

Several studies have confirmed that most corrosion processes obey a first order kinetics according to the following relationship,

$$-\log(weight\ loss) = \frac{k_1 t}{2.303} \qquad (7)$$

Therefore, plots of -log(weight loss) versus time were linear at all temperatures and at all concentrations of SHW as shown in Fig. 2. It is also known that the rate constant is inversely proportional to the half-life multiply by 0.693 (i.e, -ln2) (Eddy *et al*., 2008). Consequently, calculated values of rate constant (deduced from the slope of the plot) and half-life of aluminum in solution of HCl (in the absence and presence of SHW) are recorded in Table 3. The results revealed that various concentrations of SHW extended the half-life of aluminum in solution of HCl

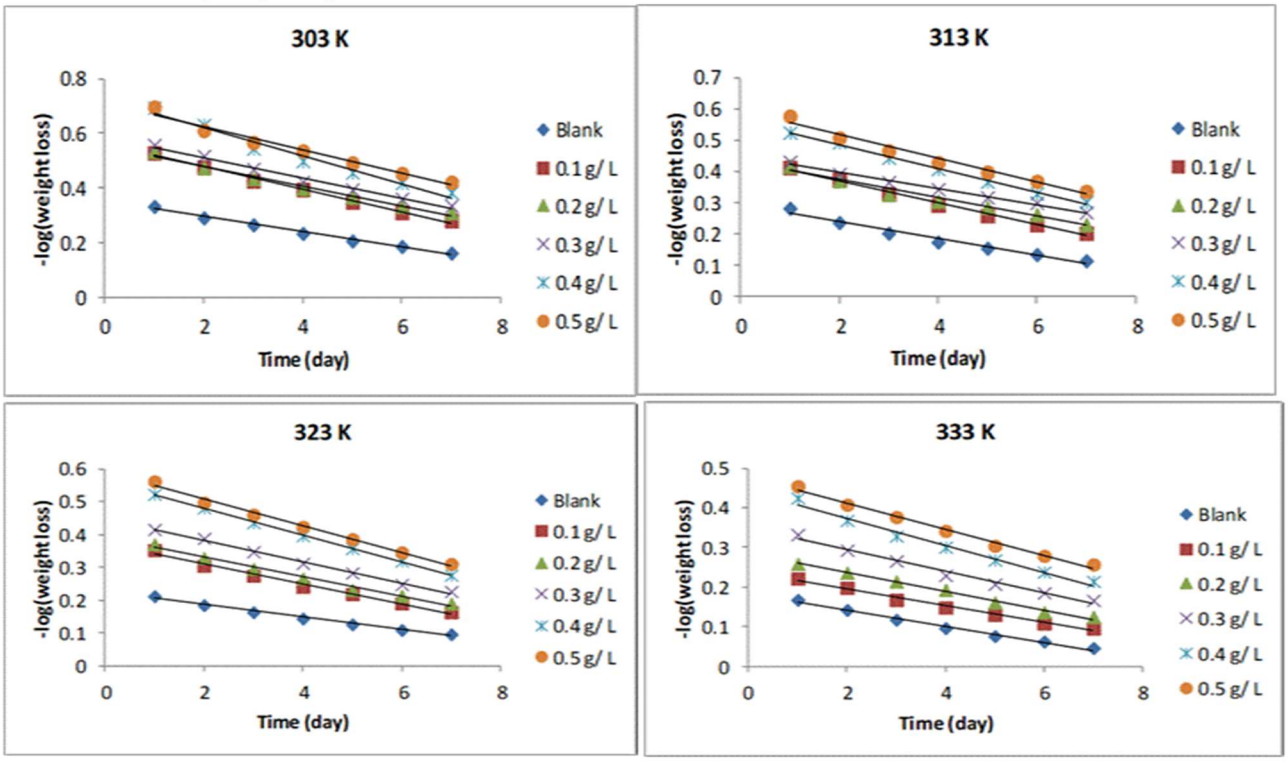

Fig. 2: Variation of –log(weight loss) with time for the corrosion of aluminium in 0.1 M HCl containing various concentrations of SHW at 303 to 333 K

Table 3: Kinetic parameters for the inhibition of the corrosion of aluminium in 0.1 M HCl by SHW

System	Slope	K_1	$t_{1/2}$ (day)	R^2
Blank (0.1 M HCl)	0.0279	0.0643	4.1	0.9910
0.1 g/L SHW at 303 K	0.0413	0.0951	5.8	0.9933
0.2 g/L SHW at 303 K	0.0365	0.0841	7.3	0.9826
0.3 g/L SHW at 303 K	0.0377	0.0868	8.0	0.9884
0.4 g/L SHW at 303 K	0.0515	0.1186	8.2	0.9730
0.5 g/L SHW at 303 K	0.0434	0.1000	10.8	0.9742
Blank (0.1 M HCl)	0.0270	0.0622	3.6	0.9715
0.1 g/L SHW at 313 K	0.0353	0.0813	7.9	0.9924
0.2 g/L SHW at 313 K	0.0293	0.0675	11.8	0.9734
0.3 g/L SHW at 313 K	0.0256	0.0590	10.3	0.9904
0.4 g/L SHW at 313 K	0.0382	0.0880	8.5	0.9963
0.5 g/L SHW at 313 K	0.0381	0.0877	11.1	0.9755
Blank (0.1 M HCl)	0.0192	0.0442	2.9	0.9890
0.1 g/L SHW at 323 K	0.0305	0.0702	7.4	0.9888
0.2 g/L SHW at 323 K	0.0299	0.0689	7.4	0.9879
0.3 g/L SHW at 323 K	0.3260	0.7508	10.1	0.9954
0.4 g/L SHW at 323 K	0.0408	0.0940	9.9	0.9995
0.5 g/L SHW at 323 K	0.0405	0.0933	15.7	0.9918
Blank (0.1 M HCl)	0.0202	0.0465	2.2	0.9888
0.1 g/L SHW at 333 K	0.0213	0.0491	8.9	0.9890
0.2 g/L SHW at 333 K	0.0233	0.0537	10.9	0.9937
0.3 g/L SHW at 333 K	0.0275	0.0633	12.9	0.9851
0.4 g/L SHW at 333 K	0.0340	0.0783	14.1	0.9822
0.5 g/L SHW at 333 K	0.0329	0.0758	14.9	0.9899

Corrosion inhibition process is facilitated by adsorption of the inhibitor on the surface of the metal, which is the initial step in the inhibition process. The minimum energy needed to activate the adsorption process can be estimated from the Arrhenius equation which can be written according to the following equation (Eddy *et al*., 2009a)

$$\ln(CR) = lnA - \frac{E_a}{RT} \qquad (8)$$

In line with equation 11, plots of ln(CR) versus 1/T were linear as shown in Fig. 3 while adsorption parameters deduced from the plots are presented in Table 4. The results indicated higher activation energy in the presence of the inhibitor than in the absence of the inhibitor (i.e the blank) indicating that the inhibitor retarded the corrosion of aluminum through adsorption. The activation energies were also within the range expected for physisorption mechanism, which align with the findings deduced from the trend of variation of inhibition efficiency with temperature (Eddy and Odoemelam, 2008).

3.3 Thermodynamics/adsorption study

The transition state equation (equation 9) was used to estimate the thermodynamic parameters associated with the adsorption of SHW on the surface of aluminum (Eddy *et al*., 2009b)

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

Consequently, enthalpy and entropy of adsorption of SHW on aluminum surface were calculated from the slope and intercept of the Transition state plots (also shown in Fig.3) generated through equation 9 presented in Table 4. Calculated values of enthalpy change are low and negative which is evident of exothermic reaction and physiosorption mechanism (Ameh and Eddy, 2018b).

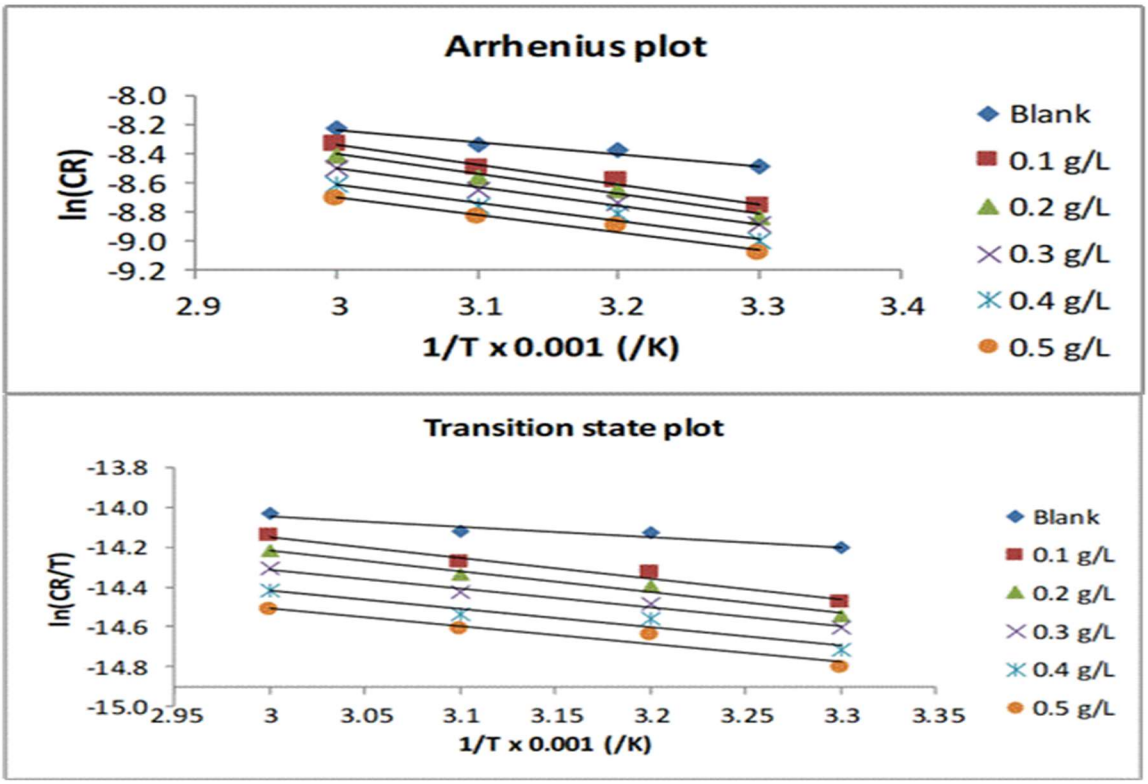

Fig. 3: Arrhenius and Transition state plots for the inhibition of the corrosion of aluminium in solution of HCl by SHW

Thermodynamic/adsorption study

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Table 4: Arrhenius and Transition state adsorption parameters for the inhibition of the corrosion of aluminium by SHW

System	Arrhenius Parameters				
	Slope	**Intercept**	**E_a (J/mol)**	**A**	**R^2**
Blank	0.8313	-5.743	6.91	3.21E-06	0.9689
0.1 g/L SHW	1.3668	-4.2411	11.36	1.44E-05	0.9842
0.2 g/L SHW	1.3634	-4.3175	11.34	1.33E-05	0.9861
0.3 g/L SHW	1.2532	-4.7465	10.42	8.68E-06	0.9936
0.4 g/L SHW	1.2278	-4.929	10.21	7.23E-06	0.9587
0.5 g/L SHW	1.1993	-5.1035	9.97	6.08E-06	0.9600
System	**Transition State Parameters**				
	Slope	**Intercept**	**$\Delta H^0_{ads}(\frac{J}{mol})$**	**$\Delta S^0_{ads}(\frac{J}{mol})$**	**R^2**
Blank (0.1 M HCl)	0.5166	-12.496	-4.30	119.75	0.923
0.1 g/L SHW	1.0522	-10.994	-8.75	132.24	0.974
0.2 g/L SHW	1.0487	-11.07	-8.72	131.61	0.9773
0.3 g/L SHW	0.9385	-11.499	-7.80	128.04	0.9887
0.4 g/L SHW	0.9131	-11.682	-7.59	126.52	0.9287
0.5 g/L SHW	0.8846	-11.856	-7.35	125.08	0.9302

Adsorption isotherms are established models for modelling the adsorption behaviour of a corrosion inhibitor depending on the nature of relationship between concentration of the inhibitor in the bulk electrolyte (C) and degree of surface coverage (θ). Tests carried out to fit adsorption data to different adsorption isotherms indicated the suitability of Temkin, Freundlich, El awardy *et al* and Dubinin-Raduskevich isotherms (Fig. 4) for the adsorption of SHW on the surface of aluminum.

The Temkin model is consistent with equation 10 and provide an interaction parameter term ('a') which were obtained through the slope of the plots (Abd El Rehim *et al*., 2016)

$$\theta = \frac{-2.303}{2a} logb_{ads} - \frac{2.303}{2a} logC \quad (10)$$

The attractive behaviour of the inhibitor is hereby proposed because the interaction parameters are positive at all concentration of SHW (Table 5).

The Freundlich adsorption model (equation 11) is significant through Freundlich adsorption parameters recorded in Table 5 (Khdom and Abod, 2016)

$$log\theta = logb_{ads} + nlog[C] \quad (11)$$

The linear plots of $log\theta$ versus $log[C]$ reveal increasing value of n with temperature and since n<1, it indicates that the adsorption is favourable while the adsorption strength decreases with temperature, a character typical for physisorption mechanism (Ye *et al*., 2019)

El awrdy *et al* plots (Fig.4) were also developed to justify the strength of adsorption according to equation 12 ,

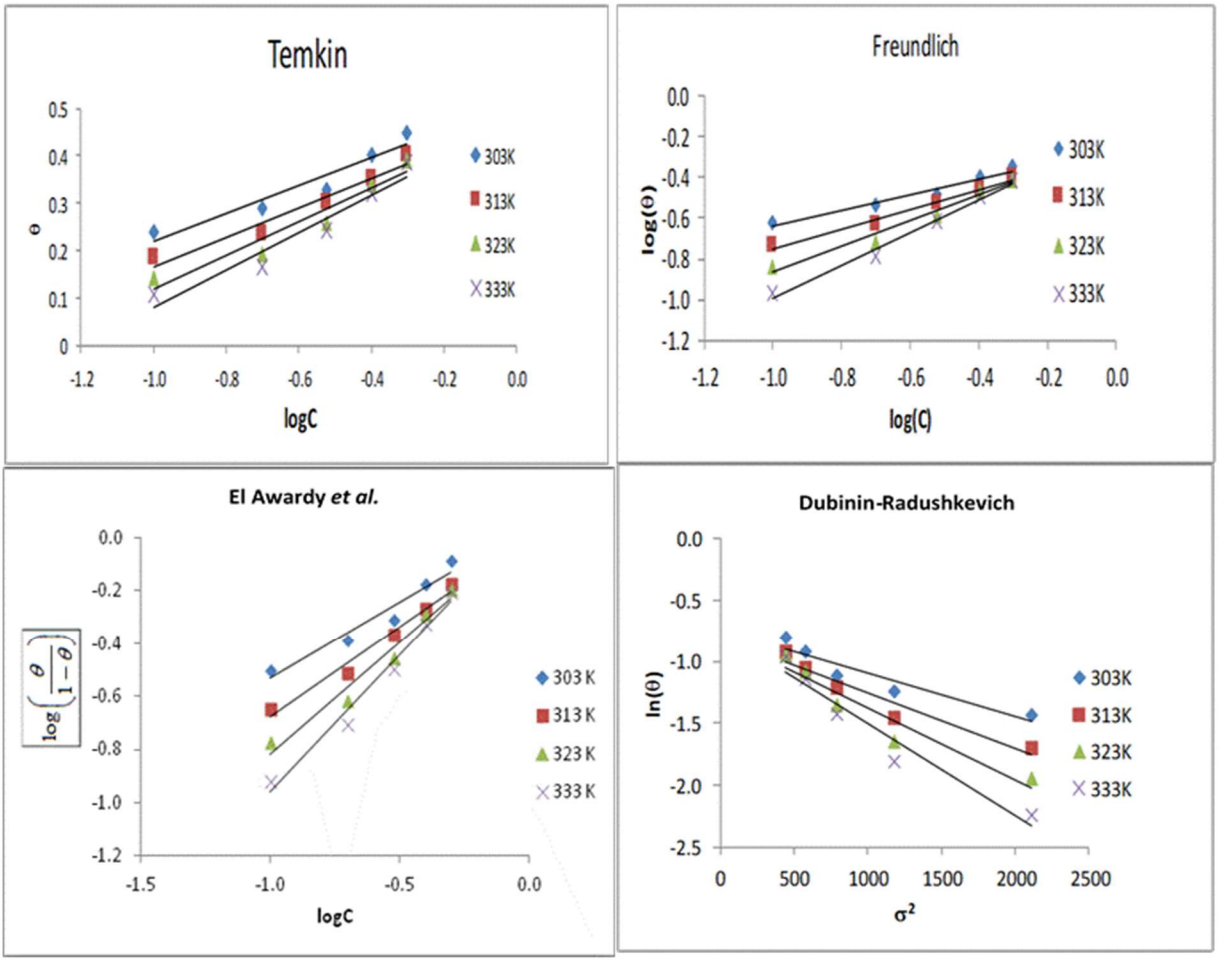

Fig. 4: Temkin, Freundlich, El Awardy *et al*. and Dubinin-Radushkevich isotherm for the adsorption of SHW on aluminum surface

Table 5: Temkin parameters for the adsorption of SHW on aluminium surface at various temperatures

Temkin parameters						
T (K)	Slope	Intercept	a	logb	$\Delta G^0_{ads}(\frac{J}{mol})$	R^2
303	0.2929	0.5131	3.93	0.0653	-10.50	0.9202
313	0.3069	0.4728	3.75	0.0630	-10.83	0.9528
323	0.3528	0.4713	3.26	0.0722	-11.23	0.9265
333	0.3936	0.4736	2.93	0.0809	-11.64	0.9327
Freundlich parameters						
T (K)		**$logb_{ads}$**	**n**		$\Delta G^0_{ads}(\frac{J}{mol})$	**R^2**
303		-0.2506	0.39		-8.64	0.961
313		-0.2635	0.49		-8.85	0.9856
323		-0.2336	0.63		-9.32	0.9771
333		-0.1839	0.81		-9.92	0.9901
El Awardy *et al*. parameters						
T (K)	**slope**	**intercept**	**1/y**	**B**	**R^2**	
303	0.5809	0.0472	1.721467	1.065181	0.9423	
313	0.6758	-0.0004	1.479728	0.999378	0.9757	
323	0.8426	0.0253	1.186803	1.05032	0.9642	
333	1.0342	0.0733	0.966931	1.19075	0.9799	
Dubinin-Radushkevich parameters						
T (K)	**'a'**	**lnx**	**x**	**E (J/mol)**	**R^2**	
303	0.0004	-0.7382	0.477973	35.36	0.8875	
313	0.0005	-0.8048	0.447177	31.62	0.8875	
323	0.0006	-0.7978	0.450319	28.87	0.9152	
333	0.0007	-0.7488	0.472934	26.73	0.9419	

The El-Awady *et al* kinetic isotherm (equation 13) can provide further insight into the strength of adsorption (Eddy *et al*., 2015).,

$$\log\left(\frac{\theta}{1-\theta}\right) = logb' + ylogC \qquad (13)$$

where y is the number of inhibitor molecules occupying one active site and 1/y represents the number of active sites on the surface occupied by one molecule of the inhibitor. 'y' is also related to the binding constant, B through $B = b'^{\left(\frac{1}{y}\right)}$. The results (Table 5) reveal that values of 1/y are above unity and tend to decrease with temperature. Hence the existent of multimolecular layer of adsorption and decreasing strength of adsorption with temperature (peculiar to physisorption) is upheld (Ukpen *et al*., 2014). The Dubinin-Raduskevich adsorption isotherm (shown in Fig.4) is consistent with the following equation,

$$\ln\theta = \ln\theta_s - a\sigma^2 \qquad (14)$$

where θ_{max} is the maximum surface coverage and σ is the polany potential and can be estimated from the following equation,

$$\sigma = RT\ln\left(1 + \frac{1}{C}\right) \qquad (15)$$

From equation 14, a plot of lnθ versus σ^2 should give a straight line (Fig.4) with slope equals to the constant, 'a', which is defined as half the square of the reciprocal of the mean adsorption energy (i.e. a = ½ $(1/E)^2$. E value less than 8 kJ/mol supports the mechanism of physical adsorption but E values greater than 8 kJ/mol are consistent with the

mechanism of chemisorption (Papoola, 2019). Therefore, physisorption mechanism is confirmed for the adsorption of SHW unto aluminum surface.

Additional information that can be derived from adsorption isotherms is the free energy of adsorption, which is related to the adsorption-desorption equilibrium constant according to equation 16 (Eddy *et al.,* 2011b)

$$\Delta G^0_{ads} = -2.303RTlog(55.5 \times b_{ads}) \quad (16)$$

The free energy change calculated through the Langmuir and Freundlich adsorption-desorption constants ranged from -10.50 to -11.64 and from -8.64 to -9.92 J/mol respectively. Therefore, the adsorption is spontaneous because ΔG^0_{ads} values are negative and support the mechanism of physical adsorption because they are negatively less than the threshold value required for chemisorption (Eddy and Odiongenyi, 2010)

4.0 Conclusion

SHW is not a very good corrosion inhibitor for aluminum but its efficiency is strongly enhanced by synergistic interaction with KBR and KCl. The inhibitor functions as an adsorption inhibitor and obeyed the adsorption models of Temkin, Freundlich, El awardy *et al* and Dubinin-Radusckevich. Data obtained for the variation of the inhibition efficiency with temperature, range of values for the activation and free energies and thermodynamic parameters confirmed thar SHW inhibited the corrosion of aluminum through the mechanism of physical adsorption.

5.0 Acknowledgement

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