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Adsorptive and Inhibitive Potential of Halide Ions and Plant Extracts from *Cucurbita Maxima* as Stainless Steel Inhibitors

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Abstract

By utilizing weight loss measurements and electrochemical technique, the corrosion inhibition efficiency of Cucurbita maxima peel and seed extracts on Grade 304 austenitic stainless steel in 1 M sulphuric acid solution was examined. The efficacy of C. maxima extracts' inhibition increased with concentration while decreasing with temperature. At 0.7 g/L extract concentration for 3 hours of immersion, the inhibitor's greatest efficiency was 95.21% for PCM and 94.26% for SCM. Stainless steel corrosion was successfully inhibited by C. maxima peel and seed extracts, according to the results, and it was discovered that the presence of halide ions enhanced the efficacy of the inhibition. The synergistic effect of halide ions was found to follow the order: KI > KCl. According to the polarization curve, C. maxima functions as a mixed-type inhibitor. Due to the extract components' adsorption on the stainless steel surface, peel and seed extracts exhibit an inhibitory effect. The activation energy of the corrosion reaction increases by the presence of both extracts. The physical adsorption is shown by the negative values of ΔG_{ads}*, which range from -12 kJ/mol to -18 kJ/mol. These values show the spontaneity of the adsorption process. Adsorption is a physical process that adheres to Langmuir adsorption isotherms. The results of a study using scanning electron microscopy to examine the surface morphology of stainless steel in inhibited and uninhibited acid solutions revealed that the presence of extract and halide inhibitors remarkably lowers the corrosion rate. This is as a result of the extract and halide inhibitor molecules adhering to the surface and forming a barrier that prevents an acid attack.*

Keywords: *Cucurbita maxima*, Stainless steel, Sulphuric acid, Weight loss, Electrochemical technique.

INTRODUCTION

To meet the challenging requirements, stainless steels are a class of adaptable materials that can be designed to display a wide range of engineering features through alloy design and regulated mechanical treatments. This adaptability has resulted in increased demand for stainless steels in a wide range of applications, including tiny pins, vehicle construction, petrochemical, space, aeronautics, shipbuilding sectors, and nuclear power plants (Dhruv, 2017). Due to its biocompatibility, certain types of stainless steel are employed in the

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production of biomedical implants. Dilute acid solutions are widely used in several industrial processes such as pickling, cleaning and descaling to remove the undesirable scales and rusts on the steel surface (Wang *et al.,* 2012).

The molecular structure of corrosion inhibitors determines their effectiveness. (Ebenso *et al.,* 2008; Rani and Basu 2012; Preethi Kumari *et al.,* 2014) have stated that organic corrosion inhibitors have heterogeneous atoms such as O, N, S, and P, which have high basicity and electron density, assisting in the corrosion inhibition of metals and alloys. Since many corrosion inhibitors threaten the environment with their toxicity even though they possess high corrosion inhibition efficiency (Ramesh and Rajeswari 2004), this sparked interest among corrosion engineers and scientists also chemists, and polymer chemists and engineers to develop a new class of inhibitors that does not or pose minimal threat to the environment and the inhibitors should have high corrosion efficiency.

Plant extracts are an extremely rich source of natural chemical compounds that may be extricated using a simple, low-cost technique and are biodegradable in nature. As a green alternative to poisonous and hazardous substances, the natural plant extract comprises an array of organic compounds such as amino acids, alkaloids, steroids, flavonoids, proteins, and tannins (Anbarasi and Vasudha 2014). In addition, these compounds have developed into corrosion inhibitor substitutes that are affordable, readily accessible, and renewable (Jokar *et al*., 2016; Chaubey *et al*., 2018; Dehghani *et al*., 2019; Dehghani *et al*., 2020).

Cucurbita maxima is a monoecious plant with therapeutic qualities in all of its parts. Pumpkin peels, which are frequently discarded, are an excellent source of vital nutrients, including dietary fiber, which slows starch digestion and improves diet-related illnesses such as diabetes (Bahramsoltani *et al*., 2017; Bai *et al.,* 2020). Pumpkin seeds, which are also tossed away, are high in oil, protein, vitamins, dietary fiber, and monounsaturated fatty acids. Nkosi *et al.* (2006) and Ashiq *et al.* (2022) discovered that these seeds have anti-diabetic, antibacterial, antioxidant, and anti-inflammatory properties. The phytochemical components of the plant have been examined, including flavonoids, alkaloids, carotenoids, steroids, saponins, carbohydrates, and amino acids. *Cucurbita maxima* peel and seed extracts can be employed as a green corrosion inhibitor (UdayaPrakash *et al.,* 2013).

Cucurbita maxima peel and seed have been utilized as a corrosion inhibitor for different metals (Anbarasi and Vasudha 2014; Anbarasi and Mini 2016). It has not, however, been studied as a potential stainless steel inhibitor in an extremely corrosive environment such as 1 M H_2S0_4. As a result, the corrosion inhibitory effectiveness of *Cucurbita maxima* peel and seed extract on stainless steel in 1M H_2S0_4 was studied in this study.

METHODOLOGY

Preparation of Coupons

Commercially purchased stainless steel was cut into coupons measuring 9 x 3cm. The coupons were polished with grade 400 emery paper, degreased in ethanol, washed with distilled water, rinsed with acetone, and dried. These plates were used for weight loss studies (Gunavathy and Murugavel 2013).

Preparation of *C. maxima* peel and seed extract

After soaking 150 g of pulverized C. maxima peel and seed in 500 ml of 95% v/v ethanol for 48 hours, the extract was obtained. A muslin cloth was used to filter the mixture first. The resulting liquid was then filtered with Whatman No. 1 filter paper, and the filtrate was concentrated using a rotary evaporator until a semi-solid extract was left. The semi-solid extract obtained was oven-dried to a solid residue at 45 ºC for 15 mins, weighed, and stored in a Bama bottle for use (Madu *et al.,* 2019). The phytochemicals (Alkaloids, tannin, flavonoids, phenol, protein, and saponin) in the extract were determined by the method reported by Sofowora (1993).

Weight loss method

Effect of Concentration and Temperature

At varying concentrations (0.3, 0.5, and 0.7 g/L), plant extracts containing alkaloids, tannins, saponins, flavonoids, proteins, and phenolic compounds were prepared. The experiment was carried out at temperatures ranging from 30 to 60 to 90ºC. The weight loss was then calculated after 3 hours of immersion. The metal coupons were taken from the solutions during the measurements, cleaned with ethanol, rinsed with acetone, and dried with paper towels. The weights were obtained and used to estimate the effect of concentration and temperature changes that occurred during the experiment (Madu *et al.,* 2019).

The extract's effect was calculated using the following equations: weight loss (Equation 1), corrosion rate (Equation 2), surface coverage (Equation 3), and percent inhibition efficiency (Equation 4).

$$\Delta W = w_2 - w_1 \quad (1)$$

$$CR(mmy) = \frac{87.6 \, x \, W}{\rho A t} \quad (2)$$

$$\text{Surface Coverage } (\theta) = \frac{W2 - W1}{W2} \quad (3)$$

$$\%IE = \left(\frac{\text{CRblank} - CRinh}{\text{CRblank}}\right) x \, 100 \quad (4)$$

Where ΔW is the mass change in mg, and w_1 and w_2 are the initial and final masses, respectively. In Equation 2, CR represents the corrosion rate in mm/y, A is the area of the stainless steel bars in cm^2, and t is the duration in hours. The coupon's weight loss in the electrolyte with the *C. maxima* inhibitor is w_1, while the coupon's weight loss in the electrolyte without the inhibitor is w_2. The surface coverage of the inhibitor on the surface of the steel is θ, w_1 and w_2 (Equation 4) are the change in mass of stainless steel in solution with inhibitor and without inhibitor, respectively (Nchewi *et al.,*2019; Madu *et al.,* 2019).

Electrochemical measurements

The computer-controlled Parstat 2273 was used for the electrochemical experiment. The Power Suite software was used to collect data, and ZsimpWin (version 3.21) was used to analyze it. A three-electrode setup was used, with platinum foil serving as the auxiliary electrode and a saturated calomel electrode serving as the reference electrode. The working electrode was a stainless steel coupon with a surface prepared according to the weight loss experimental procedure. The potentiodynamic polarization curves were recorded using the cell setup. The potentials were swept at the rate of 1.66 mVs^{-1} (Gunavathy and Murugavel 2013). The inhibition efficiency (IE) was obtained from the measured I_{corr} using the following relationship:

$$IE_p = \frac{Icorr - I'corr}{Icorr} x \, 100 \quad (5)$$

Where I'_{corr} and I_{corr} are the corrosion current density values of stainless steel in the presence and absence of inhibitors, respectively (Gunavathy and Murugavel 2013).

SEM analysis
Scanning Electron Microscope (SEM) analysis was used to study the metal surfaces after 3 hours' immersion time to understand the changes that occur before and after corrosion in the presence and absence of the extracts and halide ions using the Supra 40VP model (Madu *et al.,* 2019).

RESULTS AND DISCUSSION

Effect of concentration on Corrosion study
The study tested the weight loss method with different concentrations of *Cucurbita maxima* peel and seed extracts. Results showed that increasing the concentration increased the inhibition efficiency. At 0.7 g/L with KI in the peel, it had a maximum inhibition efficiency of 95.21%, while at 0.7 g/L with KI in the seed, it had a maximum efficiency of 94.26%. This suggests that *Cucurbita maxima* extracts act as excellent corrosion inhibitors. This is accredited to the absorption of nutrients of the extracts on the surface of the stainless steel which makes a barrier for mass and charge transfer and prevents further corrosion (Anbarasi 2016; Nchewi *et al.,* 2019; Madu *et al.,* 2019; Gunavathy and Murugavel 2013).

Effect of temperature on Corrosion study
The data in Tables 1 and 2 show that both extracts are efficient as stainless steel inhibitors in 1 M H_2SO_4 at 303 K and then decrease. A greatest inhibition of 95.21% was noted at 303 K for 0.7 g/L extract of *C. maxima* peel, while a greatest inhibition of 94.26% was observed at 303 K for 0.7 g/L extract of *C. maxima* seed. This result agrees with the works done by (Anbarasi 2016; Nchewi *et al.,* 2019; Madu *et al.,* 2019; Gunavathy and Murugavel 2013).

Effect of Halides on Corrosion study
The study found that adding KI and KCl to a H_2SO_4 solution with extracts remarkably reduced corrosion speed compared to the blank alone. When 0.5 g/L KI and KCl were added to a 1 M H_2SO_4 solution containing 0.7 g/L of extracts, weight losses were reduced, and inhibition efficiency increased. The level of inhibitory efficacy varied depending on the type of halide ion used, with the highest concentration of each halide ion having the strongest synergistic effect. The iodide has the strongest complementary effect among the two halides and thus will be focused on. This aligns with previous research done by (Ridhwan *et al.,* 2012).

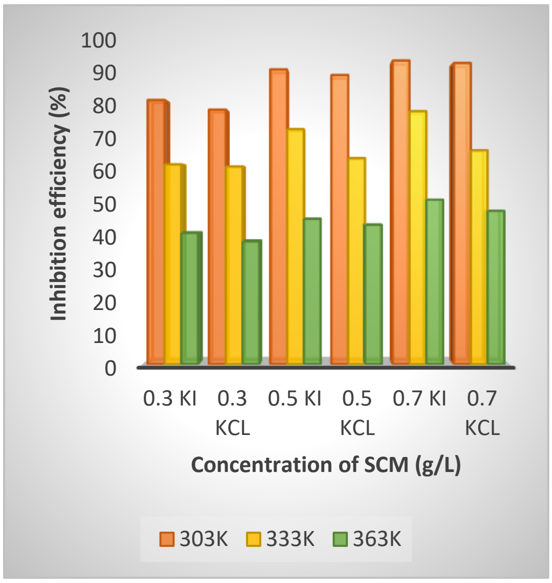
Table 1. Calculated values of Corrosion Rate, Surface Coverage, and Inhibition Efficiency of *Cucurbita maxima* Seed extract with halide ions for different concentrations and temperatures

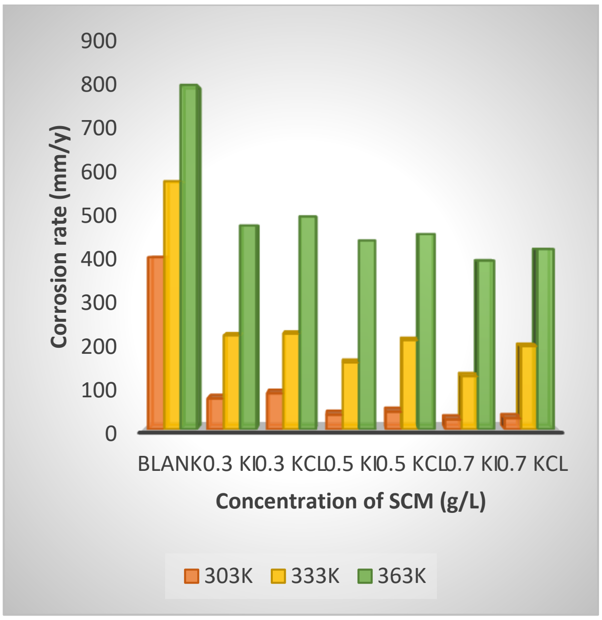
Temperature (K)	Concentration (g/L)	Δw(mg)	Corrosion rate (mm/y)	Inhibition efficiency (%IE)	Surface Coverage (θ)
303K	Blank	3664	401	-	-
	0.3 with KI	659	72	82.04	0.8204
	0.3 with KCl	768	84	79.05	0.7905
	0.5 with KI	312	34	91.52	0.9152
	0.5 with KCl	375	41	89.77	0.8977
	0.7 with KI	207	23	94.26	0.9426
	0.7 with KCl	233	26	93.51	0.9351
333K	Blank	5272	578	-	-
	0.3 with KI	1999	219	62.11	0.6211
	0.3 with KCl	2031	223	61.42	0.6142
	0.5 with KI	1425	156	73.01	0.7301
	0.5 with KCl	1901	208	64.01	0.6401
	0.7 with KI	1131	124	78.55	0.7855
	0.7 with KCl	1771	194	66.44	0.6644

363K	Blank	7316	802	-	-
	0.3 with KI	4332	475	40.77	0.4077
	0.3 with KCl	4527	496	38.15	0.3815
	0.5 with KI	4015	440	45.13	0.4513
	0.5 with KCl	4153	455	43.26	0.4326
	0.7 with KI	3589	393	51.10	0.5110
	0.7 with KCl	3834	420	47.63	0.4763

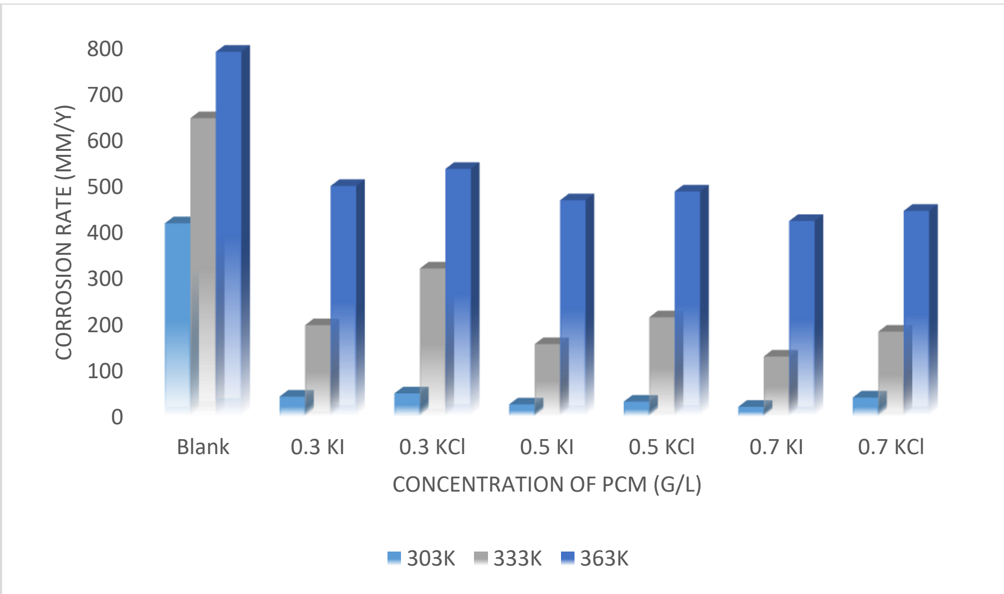
Table 2. Calculated values of Corrosion Rate, Surface Coverage, and Inhibition Efficiency of *Cucurbita maxima* Peel extract with halide ions for different concentrations and temperatures

Temperature (K)	Concentration (g/L)	Δw (mg)	Corrosion rate (mm/y)	Inhibition efficiency (%IE)	Surface Coverage (θ)
303K	Blank	3817	418	-	-
	0.3 with KI	383	42	89.95	0.8995
	0.3 with KCl	449	49	88.27	0.8827
	0.5 with KI	227	25	94.01	0.9401
	0.5 with KCl	286	31	92.58	0.9258
	0.7 with KI	186	20	95.21	0.9521
	0.7 with KCl	363	40	90.43	0.9043
333K	Blank	5896	646	-	-
	0.3 with KI	1801	197	69.50	0.6950
	0.3 with KCl	2925	320	50.46	0.5046
	0.5 with KI	1425	156	75.85	0.7585
	0.5 with KCl	1952	214	66.87	0.6687
	0.7 with KI	1175	129	80.03	0.8003
	0.7 with KCl	1674	183	71.67	0.7167
363K	Blank	7215	790	-	-
	0.3 with KI	4557	499	36.84	0.3684
	0.3 with KCl	4894	536	32.15	0.3215
	0.5 with KI	4271	468	40.75	0.4075
	0.5 with KCl	4443	487	38.35	0.3835
	0.7 with KI	3861	423	46.45	0.4645
	0.7 with KCl	4058	445	43.67	0.4367





Figures 1 and 2: Corrosion rate and inhibition efficiency of Seed extract of *C. maxima*



Figures 3: Corrosion rate of Peel extract of *C.maxima*

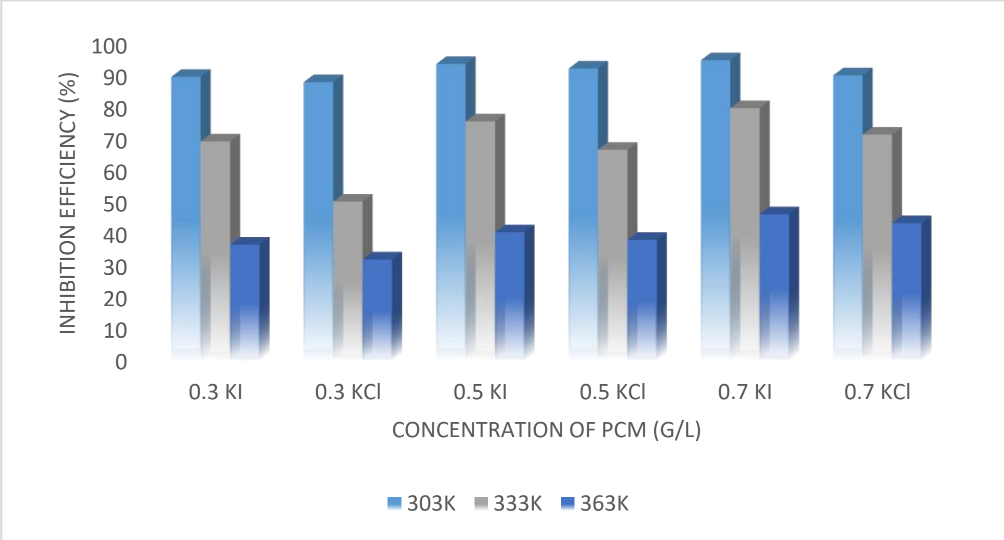


Figure 4: Inhibition efficiency of Peel extract of *C.maxima*

The inhibition efficacy and surface coverage clearly increased with increasing inhibitor concentration and decreased with increasing temperature (303-363 K). This points to the physical adsorption (physisorption) mechanism as relatable to the work of (Anbarasi and Mini 2016).

Potentiodynamic polarisation results

Table 3 lists the various electrochemical parameters determined from the Tafel diagram in Figure 5. The presence of an inhibitor results in lower corrosion current density (*Icorr*) values without producing remarkable changes in corrosion potential (*Ecorr*), indicating that the compound is a mixed-type inhibitor that is absorbed on the surface and blocks the corrosion process. In all concentrations, *βc* is bigger than *βa*, indicating that, when inhibition is under mixed control, the inhibitor's effect on anodic polarization is less pronounced than on cathodic polarization. This is related to the work of (Gunavathy and Murugavel 2013).

Table 3. Potentiodynamic polarization parameters of both extracts at different concentrations with and without KI.

Concentrations	-βa (mV dec^{-1})	-βc (mV dec^{-1})	Ecorr (mV)	Icorr (μA cm^{-2})	IE %
Blank	65.90	101.60	-491.0	77.30	-
0.3 g/L SCM	92.20	94.30	-495.0	55.80	27.81
0.3 g/L SCM + KI	63.40	165.80	-447.0	17.90	76.84
0.5 g/L SCM	81.60	122.50	-466.0	44.30	42.69
0.5 g/L SCM + KI	55.10	157.70	-427.0	12.30	84.09
0.7 g/L SCM	75.57	160.38	-459.0	29.90	61.31
0.7 g/L SCM + KI	42.40	68.50	-432.0	9.98	87.09
0.3 g/L PCM	90.10	92.30	-492.0	53.80	30.40
0.3 g/L PCM + KI	50.10	156.60	-420.0	10.60	86.29
0.5 g/L PCM	46.10	107.90	-382.0	39.00	49.55
0.5 g/L PCM + KI	44.10	149.70	-410.0	6.90	91.07
0.7 g/L PCM	73.40	155.80	-457.0	27.90	63.91
0.7 g/L PCM + KI	32.10	96.30	-393.0	4.90	93.66

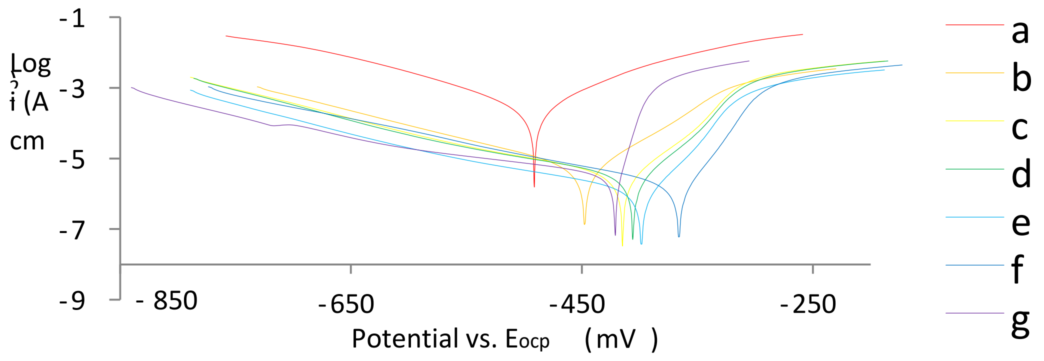


Figure 5: Polarization curves of stainless steel in 1 M H_2SO_4 in the presence of different concentrations of both extracts with KI: (a) blank; (b) 0.3 g/L SCM (c) 0.5 g/L SCM (d) 0.3 g/L PCM (e) 0.5 g/L PCM (f) 0.7 g/L PCM (g) 0.7 g/L SCM

Adsorption isotherm

The experimental results were applied to various adsorption isotherm models (Langmuir, Temkin, Freundlich, and El-Awady). For both extracts, the experimental data fit best in the Langmuir adsorption isotherm. Figures 6 and 7 show straight lines with linear regression when C/θ is plotted against C, and R^2 values of 0.999 and 1.000 are produced by this plot. This supports the hypothesis that the adsorption of the extracts in 1M H_2SO_4 solution in the presence of halide ions on the stainless steel surface follows the Langmuir adsorption isotherm, as indicated by Equation 6.

$$\frac{C}{\theta} = \frac{1}{K} + C \qquad (6)$$

Where *C* is the concentration of the inhibitor and *K* is the equilibrium constant for the adsorption process which mirrors the degree of interaction between the inhibitor and the metal surface (Saleh *et al.*, 2019).

ΔG_{ads} was calculated using the following equation:

$$\Delta G_{ads} = -RT \ln(55.5K_{ads}) \qquad (7)$$

where *R* is the molar gas constant (8.314 J/K), *T* is the temperature in Kelvin, and value 55.5 is the molar concentration of water in solution (Okewale and Adesina, 2020).

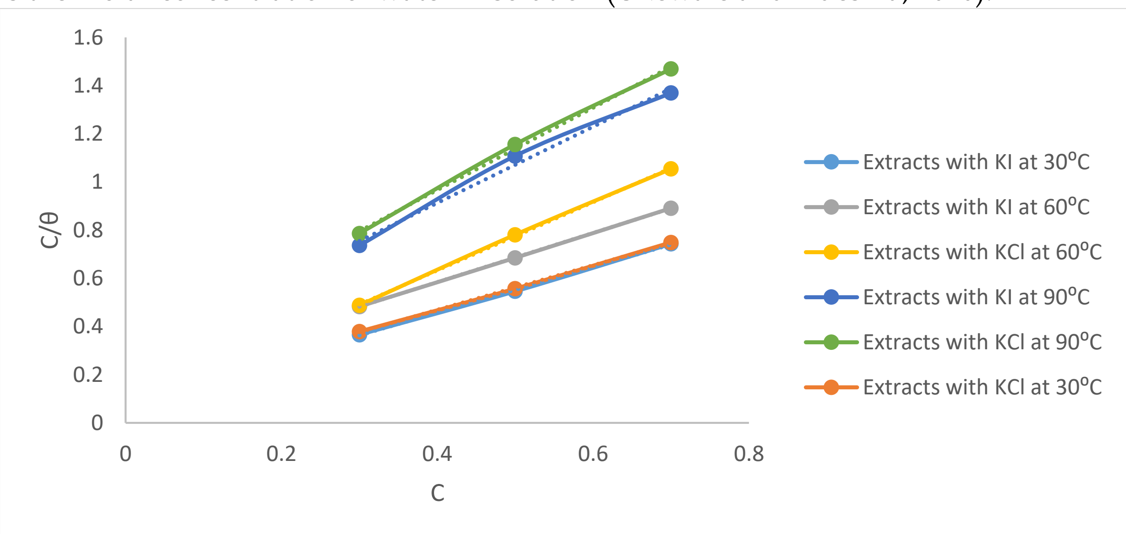


Figure 6: Langmuir isotherm for seed extract of stainless steel corrosion in 1M H_2SO_4

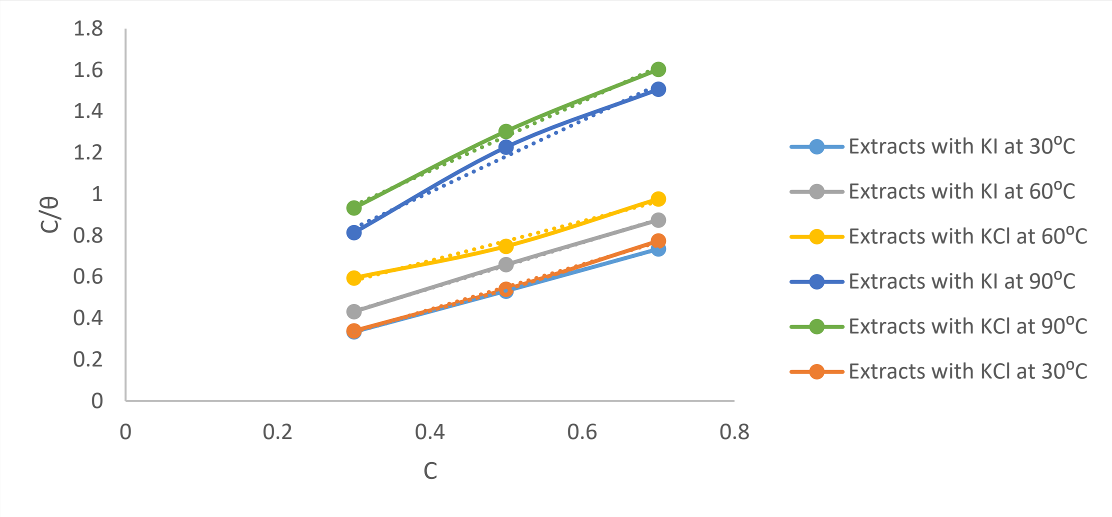


Figure 7: Langmuir isotherm for peel extract of stainless steel corrosion in 1M H_2SO_4

The plot of log CR versus 1/T produces straight lines as shown in Figures 8 and 9 which indicates the Arrhenius adsorption isotherm.

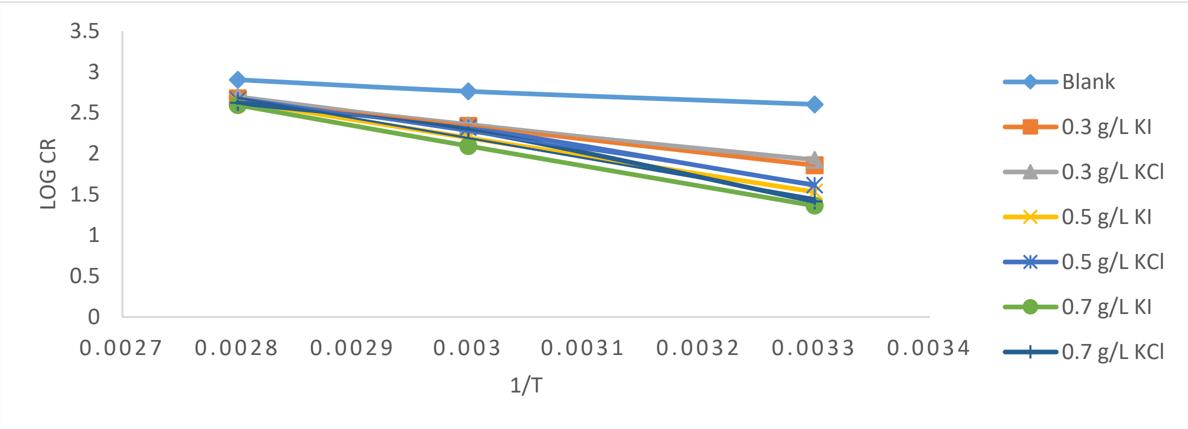


Figure 8: Plot of log CR versus 1/T for Seed extract

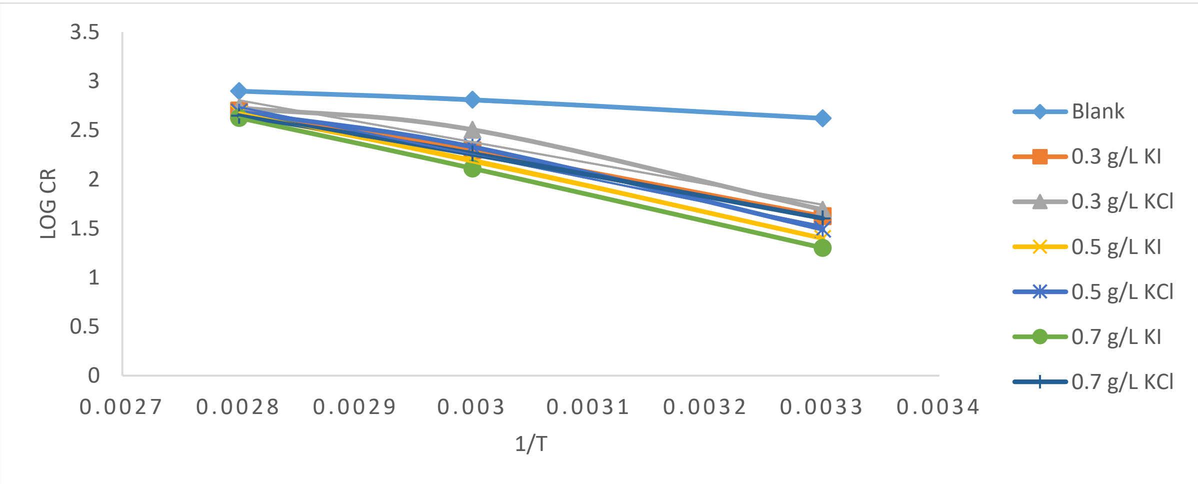


Figure 9: Plot of log CR versus 1/T for Peel extract

ΔG_{ads} computed values were negative, indicating that the adsorption process is spontaneous. Generally, the values of ΔG_{ads} less than -20 KJ mol^{-1} are congruous with the electrostatic interaction between charged inhibitor molecules and charged metal surface physical adsorption (physisorption) while values more negative than -40 KJ mol^{-1} involve sharing or transfer of electrons from inhibitor molecules to the metal surface to form a coordinate or covalent type of bond (chemisorption) (Abeng *et al.,* 2017). According to the results in Table 4, ΔG_{ads} values are less than -20 KJ mol-1. This demonstrated that the inhibitor adsorption on the metal surface is spontaneous, confirming the physisorption mechanism.

Table 4: Calculated Thermodynamic parameter ΔG_{ads} at different temperatures

The concentration of both extracts	ΔG_{ads} KJ/mol					
	303K (PCM)	333K (PCM)	363K (PCM)	303K (SCM)	333K (SCM)	363K (SCM)
0.3g with KI	-18.64	-16.72	-14.12	-16.97	-15.84	-14.64
0.3g with KCl	-18.39	-14.51	-13.55	-16.53	-15.73	-14.37
0.5g with KI	-18.90	-16.22	-13.13	-17.86	-15.78	-13.55
0.5g with KCl	-18.14	-14.95	-12.68	-17.33	-14.67	-13.34
0.7g with KI	-18.64	-15.95	-12.68	-18.04	-15.70	-13.30
0.7g with KCl	-16.68	-14.67	-12.40	-17.71	-13.98	-12.92

The thermodynamic characteristics of the corrosion process, such as enthalpy (ΔH_{ads}) and entropy (ΔS_{ads}), were calculated using the transition state theory equation given by equation 8 (Ogoke *et al.,* 2009; Mouheddin *et al.,* 2018)

$$\text{Log}\left(\frac{CR}{T}\right) = \left[\text{Log}\left(\frac{R}{Nh}\right) + \frac{\Delta S^\circ}{2.303R}\right] - \frac{\Delta H^\circ}{2.303RT} \quad (8)$$

Where R is the Universal gas constant (8.314J/Kmol), N is Avogadro's number, (6.022×10^{23} mol^{-1}), h is the Planck's constant (6.626176×10^{-34}Js) and T is the temperature of the medium. Figures 8 and 9 show a linear plot of log CR against 1/T, from which (ΔH^o) and (ΔS^o) values were computed from the slopes and intercept of the graph respectively and are presented in Table 5. According to Table 5, the blank activation energies of PCM and SCM are lower than when the inhibitors with KI are present. This apparent increase in activation energies for stainless steel dissolving in the presence of an inhibitor could be attributable to a physical adsorption process. The trend of increasing Ea values as with inhibitor concentrations has been reported by other researchers on studies on various plant extract such as black pepper (Quraishi *et al*., 2009), sunflower leaves (Cang *et al*., 2012), jujube leaves (Shivakumar and Mohana, 2012), and piper nigrum extract (Norzila and Anis, 2015). The positive sign of the ΔH^o shows that the stainless steel is endothermic in the presence of both extracts. The activation entropy (ΔS^o) was positive in the absence and presence of both extracts with halide extract inhibitor. This can be taken to infer that the activated complex represents a dissociation step rather than an association step in the rate-determining phase, which suggests that throughout the adsorption process, an increase in degree of orderliness occurs as it moves to the activated complex from the reactants (Nooshabadi and Ghandchi 2015).

Table 5: Calculated Activation Energy and Thermodynamic parameters

Concentration of Inhibitor	Ea (kJ mol^{-1}) PCM	ΔH° (kJ mol^{-1}) PCM	ΔS° (J mol^{-1}K^{-1}) PCM	Ea (kJ mol^{-1}) SCM	ΔH° (kJ mol^{-1}) SCM	ΔS° (J mol^{-1}K^{-1}) SCM
Blank	10.72	4.66	37.21	11.42	4.96	37.96
0.3g with KI	41.30	17.93	72.73	31.36	13.62	60.35
0.3g with KCl	40.75	17.69	72.87	29.33	12.74	57.94
0.5g with KI	48.87	21.22	81.72	42.56	18.45	73.69
0.5g with KCl	46.45	20.17	79.21	40.41	17.55	71.49
0.7g with KI	50.81	22.06	83.66	47.14	20.47	78.85
0.7g with KCl	40.22	17.46	71.02	47.00	20.41	79.44

Morphological Properties of Corrosion Study using SEM analysis

SEM analysis of selected samples revealed severe corrosion on blank sample plates, with the austerity of corrosion agents increasing with temperature. The images confirmed the metal's corrosion rates in 1 M H_2SO_4 solution at 30 ºC after 3-hour immersion time. The stainless steel surfaces were inhibited against corrosion and smoother surfaces appeared with increasing inhibitor concentration as shown in the micrographs plate b and c. This aligns with previous research done by Nchewi *et al.,* (2019) and Madu *et al.* (2019).

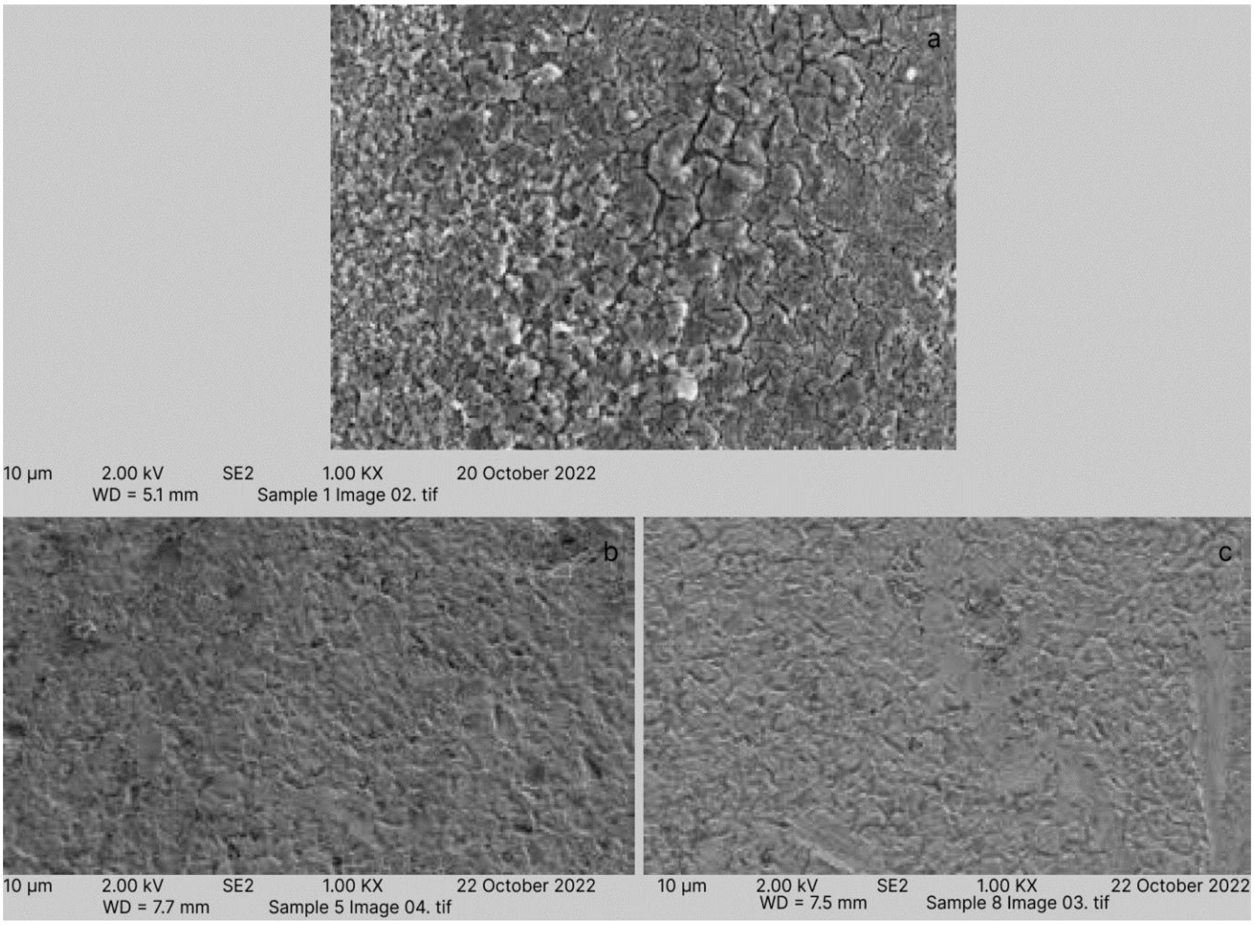


Plate 1: SEM images of stainless steels after corrosion in 1 M H_2SO_4 and with both extracts at 303 K (a) Blank (b) 0.7 g/L SCM (c) 0.7 g/L PCM

CONCLUSION

Cucurbita maxima peel and seed act as green corrosion inhibitors for stainless steel in 1M H_2SO_4 solution. With increased extract concentration, the IE rises. *C. maxima* functions as a mixed type of inhibitor, according to the potentiodynamic polarization curves. Adsorption is a physical process that follows the Langmuir adsorption isotherm. Surface experiments using SEM validated the plant extracts' effectiveness as a corrosion inhibitor for stainless steel.

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