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Industrial application and inhibition properties of *Cucurbita maxima* on metal corrosion in aggressive medium

K Anbarasi
Assistant Professor of Chemistry, Nirmala College for Women, Coimbatore, Tamil Nadu, India

Abstract

The influence of peel of *Cucurbita maxima* (PCM) on the corrosion inhibition of mild steel in 1N HCl was studied using phytochemical screening, weight loss method, polarization and FTIR studies. The results revealed that PCM was a green corrosion inhibitor of mixed type and electrochemical measurements showed maximum inhibition efficiency (IE) of 85% at and weight loss method shows the maximum IE of 97% at 2% PCM concentration. The inhibitive effect of the plant extract could be attributed to the presence of phytochemical constituents in the plant extract which is adsorbed on the metal surface. The isolated phenols and flavonoids are may be the responsible for the reduction of corrosion rate of mild steel. The EDX results attributed to the adsorption of plant extract onto the metal surface that inhibits the oxidation and corrosion. The system was found to obey El-Awady, Freundlich and Temkin isotherms.

Keywords: *Cucurbita maxima*, mild steel, corrosion inhibitor, flavonoids, polarization

1. Introduction

The deterioration of surface of the metal caused by the reaction with the environmental conditions is called corrosion. Corrosion can cause terrible damage to metal structure causing economic consequences in terms of product losses and environmental pollution. Plants contain variety of secondary metabolites compounds which are used in metal corrosion protection process, as possible replacement of toxic synthetic inhibitor. Plant products are inexpensive, biodegradable, easily obtainable and ecofriendly materials. The use of green inhibitors also has the potential of being cost effective due to the renewability of its resources [1]. Many researchers reported Natural compounds as corrosion inhibitors for metals in aggressive environments [2-5]. Many plant materials such as *Solanum tuberosum* [6], *Microdesmis puberula* [7], *Nyctanthes arbortristis* [8], *CitrusAurantifolia* [9], red onion skin [10], *Calotropis procera* [11], *Morinda Tinctoria* [12], Beet Root Extract [13], Medicago Sative plant [14], *Heinsia crinata* [15], *Ephedra alata* [16], *Juniperus* plants [17], *Mollugo cerviana* [18] and *Zingiber Officinal Roscoe* [19] used as green corrosion inhibitors for metallic corrosion process.

This study aims to investigate the mild steel corrosion in 1N HCl solution in the presence of peel of *Cucurbita maxima* (PCM) as a corrosion inhibitor using phytochemical screening, weight loss and electrochemical measurements and FTIR analysis.

2. Experimental

2.1 Materials

The mild steel sheet of 2 mm in thickness was mechanically press cut into 5 cm × 2 cm coupons. The aggressive solution of 1N HCl was prepared by dilution of analar HCl using distilled water. Stock solution of PCM extract was prepared by boiling weighed amounts of the dried peel material for 3h in 1N HCl. The solution was cooled and then filtered and stored. The concentration of the inhibitor solutions varied from 0.01% v/v to 2% v/v.

2.2 Methods

2.2.1 Phytochemical investigation

Qualitative and quantitative phytochemical study of the crude acid extract of PCM was determined according to the standard procedures to identify the constituents from the plant extract [20]. Dragendorff test for alkaloids, Foam test for saponins, Salkowski test for triterpenoids, $FeCl_3$ test for tannins, NaOH test for glycosides, Molisch test for reducing sugars, Biuret test for proteins, and ammonia test for detection of flavonoids are studied to identify the constituents present in the extract of the peel of the plant.

2.2.2 Weight loss method

The weight loss method is used to measure the corrosion rate of the metal. Weight loss measurements were conducted under total immersion of already weighed mild steel coupons in 100ml of the test solution containing 0.01,0.05%,0.1%, 0.3%, 0.5%, 0.8%, 1%,1.5% and 2% peel of *Cucurbita maxima* (PCM)) extract and in blank solution for different temperature (303K,313K,323K,333K &343K) at 1h. After the specified time, the specimens were washed immediately with distilled water, dried and weighed. From the weight loss measurement, the corrosion rate (mpy) and inhibition efficiency (IE) are calculated using the formula [21].

$$\text{Corrosion Rate (CR) (mpy)} = 534\ W/\ D\ A\ t \qquad (1)$$

Where W is the weight loss in mg, D is the density of mild steel ($7.8/cm^3$), A is the area of specimen and t is the time of immersion.

$$\text{Inhibition Efficiency (IE \%)} = [CR_{blank} - CR_{inhi} / CR_{blank}] \times 100 \qquad (2)$$

Where CR_{blank} and CR_{inhi} are the corrosion rate values in absence and in presence of inhibitor.
The effect of temperature was used to explain the kinetic and adsorption properties of the plant extract.

2.2.3 Electrochemical study

The electrochemical cell is a three-electrode Pyrex glass cell. The mild steel specimen was inserted in Teflon holder using epoxy resin with an exposed area of 0.283 cm^2. A platinum wire was used as a secondary electrode. The reference electrode (Saturated Calomel Electrode) coupled to a capillary whose tip was located between the working electrode and the secondary electrode. The electrochemical measurements along with EIS were carried out with IVIUM make potentiostat connected with a computer. The OCP was measured for 30 min before starting the EIS and it was carried out at the open-circuit potential from 10 kHz to 0.01Hz frequency range with signal amplitude of 0.025 mV and scan rate of 1 mV/s. From the polarization study the calculation of inhibition efficiency by Tafel method and LPR method are given below [22].

$$IE\ \% = [\ (I_{corr(blank)} - I_{corr(inhibitor)}) / I_{corr(blank)}\ ] \times 100 \qquad (3)$$
$$IE\ \% = [(Rp_{(inhibitor)} - Rp_{(blank)}) / Rp_{(inhibitor)}] \times 100 \qquad (4)$$

From EIS study the inhibition efficiency from Rct and cdl values are given by the following equations

$$IE\ \% = [(Rct_{inhibitor} - Rct_{blank}) / Rct_{inhibitor}\ ] \times 100 \qquad (5)$$
$$IE\ \% = [(Cdl_{blank} - Cdl_{inhibitor}) / Cdl_{blank}] \times 100 \qquad (6)$$

2.2.4 FTIR technique

The IR spectrum was performed by using a Shimadzu spectrometer in the spectral region between 4000 and 500 cm$^{-1.}$ The isolated PCM-F and PCM-P from the peel extract were employed for FTIR analysis.

2.2.5 SEM and EDX analysis

The surface morphology of the mild steel specimens were tested at Karunya University, Coimbatore using Shimadzu make scanning electron microscope in the magnification range of 500 µm. EDX spectroscopy has been used to determine the elements present on the metal surface before and after exposure to the inhibitor solution.

2.2.6 Industrial application

Field testing

Electroplating is extensively used in various industries for coating metal items with a thin layer of a different metal and increases life of metal and prevents corrosion. Corrosion inhibitors are extensively used in industry to reduce the corrosion rate of metals and alloys in contact with aggressive environment. The peel extract of *Cucurbita maxima* was tested as pickling inhibitor in local electroplating industry (Electro Finishers, Chettipalayam road, Pappampatti, Coimbatore). The quality of the electroplated samples after pickling with plain commercial hydrochloric acid (11N) and with 1N HCl containing 5% of the peel extract were tested. During the pickling process the change in hydrogen evolution and concentration of the effluent also noted.

3. Results and Discussions

3.1 Phytochemical study of PCM extract

The phytochemical screening is performed at T.stanes, Phyto Pharma Testing Lab, Coimbatore. Results of the phytochemical constituents of PCM in 1N HCl was reported in Table 1. The qualitative phytochemical screening revealed that the presence of flavonoids, glycosides, proteins, phenolic compounds and tannin and the absence of saponins, steroids, terpenoids, carbohydrates and alkaloids in the plant extract. From the quantitative analysis 60 mg of flavonoid and 10 mg of phenols are isolated from 20ml of the PCM extract. The isolated compounds are named as PCM-F (PCM-Flavonoid) and PCM-P (PCM-Phenol).

Table 1: Quantitative phytochemical screening of *Cucurbita maxima*

Sample	Phytoconstituents (Qualitative analysis)	Isolated Compound (Quantitative analysis)	
		Total Flavonoid (PCM-F)	Total Phenol(PCM-P)
Peel of *Cucurbita maxima* (PCM) in 1N HCl	Flavonoids, Tannin, Protein, Glycosides and Phenol.	60mg / 20 ml	10mg / 20 ml

3.2 FTIR Analysis

The type of bonding for organic inhibitors adsorbed on the metal surface is determined by FTIR analysis. FTIR spectra of the isolated compounds from the peel extract are shown in Fig 1 & 2. The observed stretching frequency and the corresponding band assignment are given in Table 2. It revealed that in both PCM-F and PCM-P the bands at 3365 and 3263 cm-1 indicate the presence of OH in alcohols or phenols (H Bonded). The bands at 3159 and 3169 cm-1 are attributed to the C-H stretching vibrations in aromatics. The bands at 1604, 1463 &1608, 1462 cm-1 are attributed to C=C in aromatics. The band at 1404 cm-1 indicates the presence of C-O-H bending vibrations. The band at 1078 cm-1 is attributed to C-O stretching vibration. The peaks at 725 and 723 cm-1 are assigned to C-H in ortho disubstituted benzene. The peaks at 551, 599 and 630 cm-1 are attributed to C-H bending vibration in benzene [23-26]. It is also evident that the isolated compounds are responsible for reduction of corrosion rate of the mild steel in acid media.

Table 2: FTIR peak values for Isolated compounds from PCM extract.

S. No	Frequency(cm^{-1}) for Isolated Compounds PCM-F	Frequency(cm^{-1}) for Isolated Compounds PCM-P	Band assignment
1	3365 & 3263	3365 & 3263	OH in alcohols or phenols (H Bonded)
2	3159	3169	C-H stretch in aromatics
3	1604 & 1463	1608 & 1462	C=C in aromatics
4	1404	1404	C-O-H bending vibration
5	1078	1078	C-O stretching vibration
6	725	723	C-H in ortho disubstituted benzene
7	630 & 551	599 & 551	C-H bending vibration in benzene

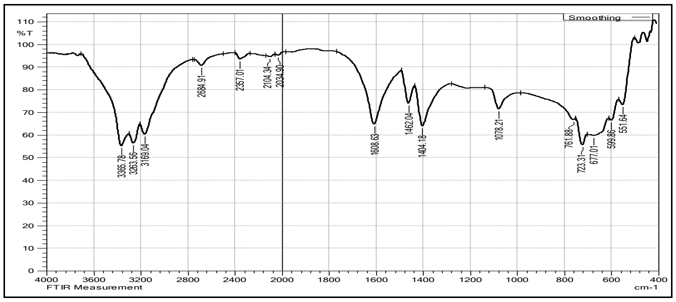


Fig 1: FTIR Spectrum of isolated PCM-P

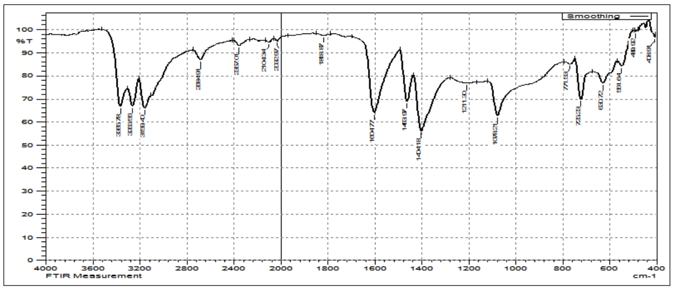


Fig 2: FTIR Spectrum of isolated PCM-F

3.3 Weight loss method

The weight loss method of screening corrosion rate and inhibition efficiency is useful because of its simple application and high reliability. The non electrochemical technique of weight loss is used to determine the corrosion rate (CR) of metal and inhibition efficiency (IE %) of tested solution. The weight loss measurements showed that there is a reduction in the corrosion rate of mild steel even for a small concentration of 0.01% of PCM. But at the same time, it can be noted that CR increases with rise in temperature. The raise in corrosion rate can be accredited to significant desorption of the inhibitor from the mild steel surface with increased temperature range. The inhibition efficiency (IE) increases with increase in concentration of inhibitor and reached the maximum value of 97% for 2% concentration of PCM at 343K. The above inspections indicate that corrosion inhibition is due to adsorption of inhibitor constituents at metal/ solution interface. The increase in inhibition efficiency and decrease in corrosion rate may be due to adsorption and desorption phenomena. The corrosion rate of mild steel and inhibition efficiency of various inhibitor concentrations at different temperature ranges are given in Table 3.

Table 3: CR of mild steel and IE of PCM in 1N HCl at different temperatures

S. No	Conc. of PCM (% v/v)	303 K		313 K		323 K		333 K		343 K	
		CR mpy	IE %	CR mpy	IE %	CR mpy	IE %	CR mpy	IE %	CR mpy	IE %
1	Blank	1204	-	2228	-	3428	-	3916	-	4453	-
2	0.01	427	65	602	73	911	73	1042	73	1705	62
3	0.05	314	74	362	84	541	84	759	81	1138	74
4	0.1	249	79	301	86	401	88	423	89	715	84
5	0.3	166	86	205	91	253	93	222	94	331	93
6	0.5	157	87	157	93	205	94	170	95	253	94
7	0.8	140	88	126	94	153	95	144	96	196	95
8	1	122	90	118	95	140	96	135	97	174	96
9	1.5	87	92	109	95	118	97	118	97	157	96
10	2	83	93	96	96	100	97	118	97	144	97

3.4 Adsorption study

The mode of interaction between the PCM inhibitor and the metal surface was studied by applying adsorption isotherms. To identify the mechanism of corrosion inhibition, the adsorption behavior of the extract adsorbents on the metal surface must be known. The predominant adsorption mode will be dependent on factors such as the extract composition and the nature of the surface charge on metal. There are a number of mathematical expressions having thus developed to take into consideration of non-ideal effects. The most used isotherms are, Frumkin, Parsons, De Boer, Flory-Huggins, Temkin and Bockris-Swinkless [27-28].

The various adsorption isotherms employed for this study are given below

Freundlich $\log \theta = \log K + n \log C$ (7)
El-Awady $\log [\theta/1-\theta] = \log K + y \log C$ (8)
Temkin $\log \theta = \log K + \log C$ (9)

The plot of log θ Vs log C is shown in Fig 3. The linearity shows that the adsorption of the inhibitor on mild steel surface obeys Freundlich isotherm. Figure 4 confirms that the inhibition processes due to adsorption of the PCM extract on the MS surface. This is for the reason that a straight line is obtained when log (θ/1-θ) is plotted against log C and the linear correlation coefficient (R^2) of the fitted data is closely related to 1. The plot of Log C Vs θ shows that the adsorption of PCM on mild steel also obeyed the Temkin isotherm (Fig 5). The data obtained best fitted the Freundlich, El-Awady and Temkin adsorption isotherm models existing by their values of coefficient of determination, R^2, existing close to unity in each case (Table 4).

Table 4: Linear regression coefficient of various adsorption isotherms for adsorption of PCM on the mild steel surface

Temperature	Linear Regression Coefficient(R^2)		
	Temkin	El-Awady	Freundlich
303K	0.987	0.982	0.978
313K	0.960	0.993	0.944
323K	0.951	0.993	0.934
333K	0.932	0.976	0.921
343K	o.934	0.980	0.917

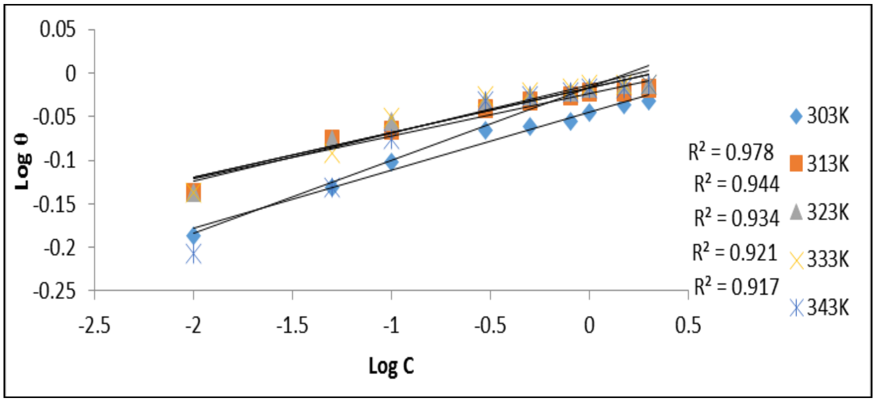


Fig 3: Freundlich plot

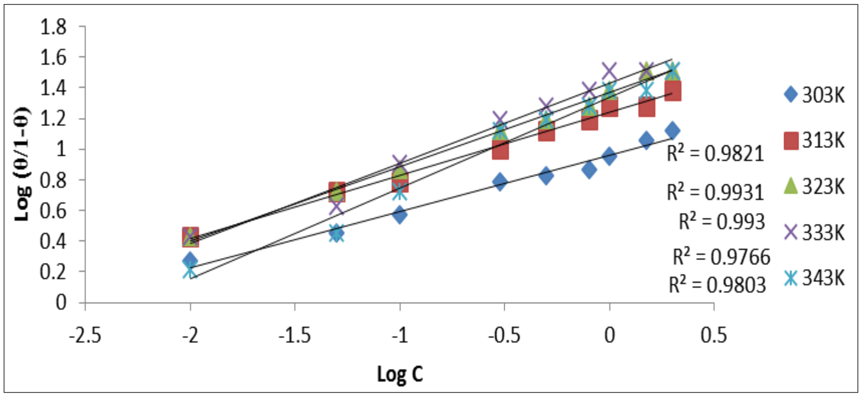


Fig 4: El-Awady plot

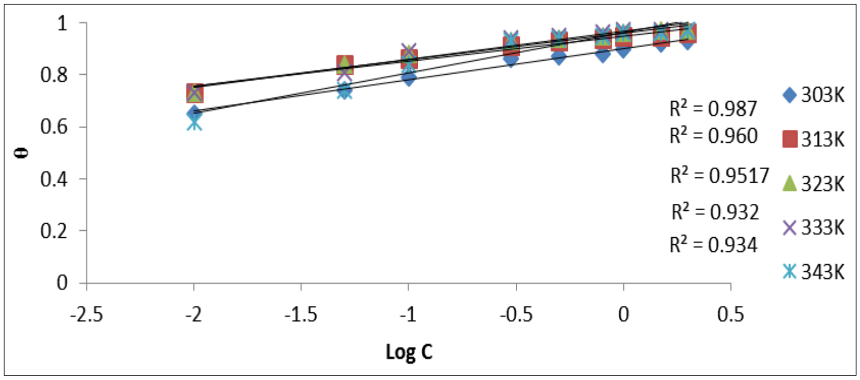


Fig 5: Temkin plot

3.5 Polarization study

The impedance parameters and electrochemical corrosion parameters like corrosion potential (E_{corr}), corrosion current density (I_{corr}), Tafel constants βa and βc are calculated from the polarization curves of mild steel in the absence and presence of various concentrations of PCM in 1N HCl and summarized in Table 5. The I_{corr} values were decreased with increased concentration of the inhibitor which indicates that the corrosion process is directed by the addition of PCM. This proves the mixed mode of inhibition of the extract. The values of both anodic and cathodic Tafel constants βa and βc respectively are remarkably changed in the presence of the extract. E_{corr} and I_{corr} values indicate the PCM inhibitor acts as a mixed type inhibitor. The increased linear polarization (R_p) value also proves the corrosion inhibitive nature of the plant extract. The calculated inhibition efficiency values showed that % IE increases with increasing PCM concentration. The maximum inhibition efficiency observed at 69% for 2% PCM concentration from LPR method. The Tafel plot was shown in Fig 6.

Table 5: Polarization and Impedance parameters for mild steel in 1N HCl in the absence and presence of PCM extract

Conc.of PCM % v/v	E_{corr} V	I_{corr} Amp/cm^2	βa V/dec	βc V/dec	R_p Ohm	IE %		Cdl (F) ×10^{-5}	Rct (Ohm)	IE %	
						Tafel	linear			Cdl	Rct
Blank	-0.4836	0.0001	0.058	0.12	106.9	-	-	3.68	15.29	-	-
0.05	-0.4758	0.0001	0.06	0.139	163.9	31	35	2.61	18.63	29	18
0.1	-0.4716	0.0007	0.059	0.14	239.1	53	55	2.90	26.77	21	42
0.3	-0.4632	0.0009	0.062	0.153	203.4	41	47	2.75	36.51	25	58
0.5	-0.4661	0.0008	0.061	0.162	218.6	45	51	2.68	47.88	27	68
0.8	-0.4485	0.0005	0.059	0.156	352.2	67	69	2.60	53.81	29	71
1	-0.4648	0.0007	0.069	0.176	297.9	55	64	2.52	70.49	32	78
2	-0.4504	0.0007	0.074	0.185	346.1	58	69	2.12	106.48	42	85

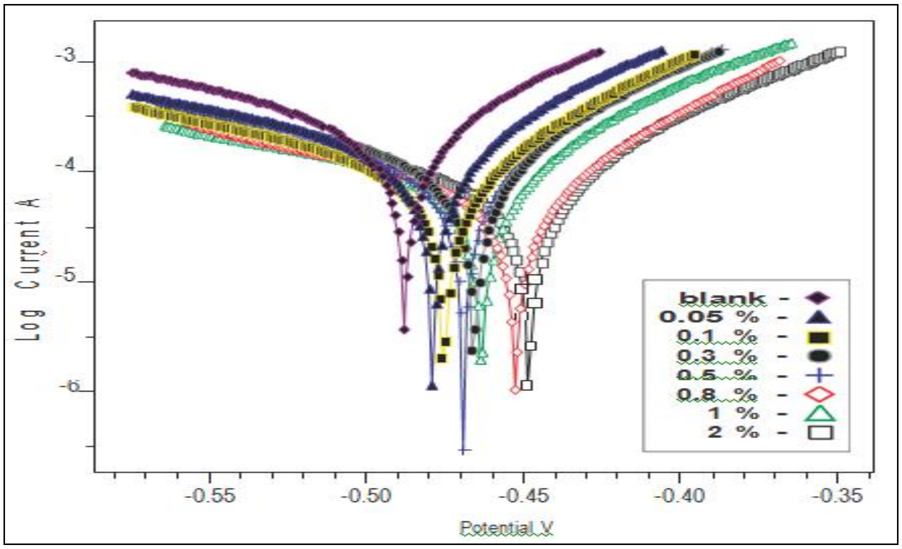


Fig 6: Tafel curve for mild steel in 1N HCl solution in absence and presence of PCM

3.6 Electrochemical Impedance study

Nyquist plots of mild steel in blank and inhibited acidic solutions containing various concentrations of PCM inhibitor shown in Fig 7. These plots indicate that the impedance response of mild steel is considerably changed by the addition of the PCM. Impedance diagrams show a semi circle appearance, indicates the mild steel corrosion mainly controlled by charge transfer process. From the impedance data, it is clear that an increase of the charge transfer resistance and decrease of the double layer capacitance (C_{dl}) with increased inhibitor concentration indicate that PCM inhibits the corrosion rate of mild steel by an adsorption mechanism. Data in table 4 indicates that the IE increased with increasing concentration of PCM. This implies that the molecules of the PCM inhibitor functioned through an adsorption process. The maximum inhibition efficiency obtained at 85% at 2% PCM concentration.

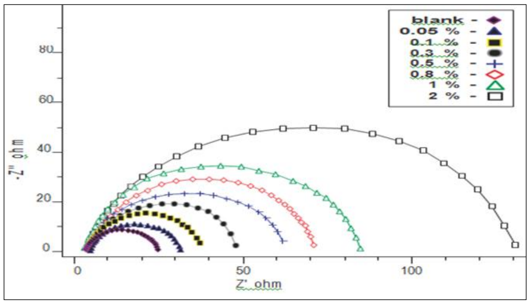
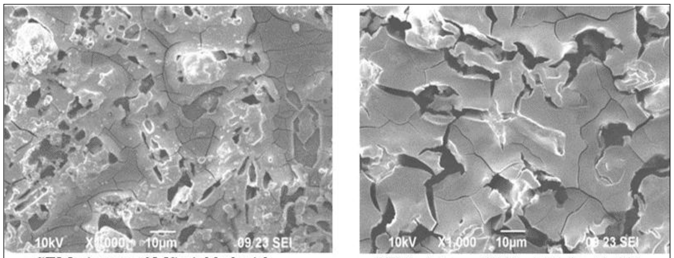


Fig 7: Nyquist plot for mild steel in 1N HCl in absence and presence of PCM

3.7. Industrial application of PCM

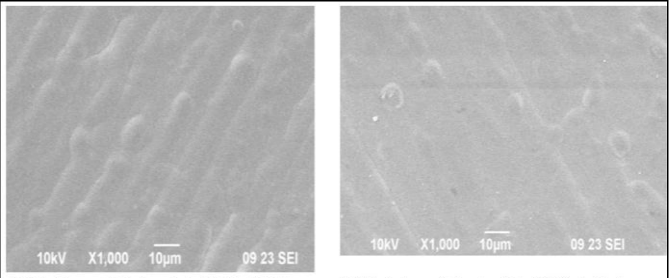
The addition of plant extract to the pickling process is viewed as "ecofriendly" compared with acid pickling and it imparts to mild steel a high degree of rust resistance, and acts as a barrier to oxidation for acid-pickled mild steel. The peel extract of *Cucurbita maxima* was employed for pickling process in electroplating industry. It showed better result compared to commercial acid pickling. The SEM and EDX analysis showed the efficiency of the plant extract. The one set of mild steel specimens immersed in commercial hydrochloric acid and another set metal specimens immersed in 1N HCl containing 5% of PCM extract for 5 minutes. It is observed that in the pickling process with commercial acid, large amount of hydrogen gas evolved but with 5% PCM the hydrogen liberation was very less and controlled. So on a large scale, pickling with1N HCl containing plant extract will be ecofriendly with less fumes and less corrosive effluents. The two sets of pickled mild steel specimens were electroplated in the local electroplating industry.

Table 6 shows the comparison of EDX analysis of mild steel samples pickled with commercial acid and 1N HCl containing 5% PCM and the comparison of EDX analysis of electroplated mild steel samples after pickled with commercial acid and 1N HCl containing 5% PCM is listed in Table 7. The SEM and EDX pictures are shown in Fig 8 and Fig 9 respectively. The SEM picture of mild steel specimen pickled with 1N HCl containing 5% PCM gives smooth and polished surface compared that with given by commercial acid pickling. The EDX spectra showed the weight percentage of iron in the sample pickled with plant extract is 54.35% and the sample pickled with commercial acid is 43.62%. Effluents can be made less acidic by using diluted acid instead of concentrated commercial acid and increasing the immersion periods. The SEM and EDX analysis of commercial acid and PCM extract in industrial pickling process proved that the additions of plant extract minimize the corrosion rate of mild steel considerably.



SEM picture of MS pickled with commercial acid

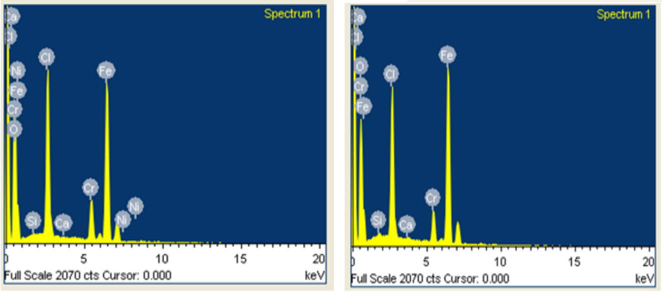
SEM picture of MS pickled with 5% PCM



SEM picture of electroplated MS pickled with commercial acid

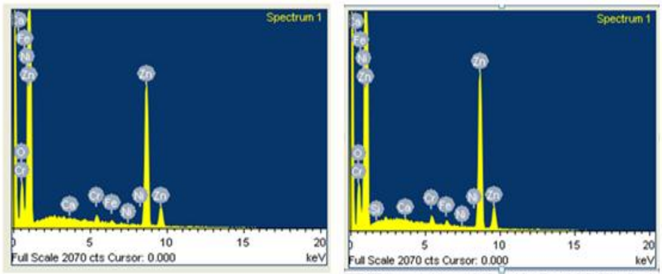
SEM picture of electroplated MS pickled with 5% PCM

Fig 8: SEM picture of mild steel samples pickled and electroplated with commercial acid and 5% PCM



EDX spectrum of MS pickled with commercial acid

EDX spectrum of MS pickled with 5% PCM



EDX spectrum of electroplated MS after pickled with commercial acid

EDX spectrum of electroplated MS after pickled with 5% PCM

Fig 9: EDX spectra of mild steel samples pickled and electroplated with commercial acid and 5% PCM extract

Table 6: Comparison of EDX analysis of mild steel samples pickled with commercial acid and 1N HCl containing 5% PCM.

Element	Pickling with Commercial Acid				Pickling with 5% PCM				
	App. Conc	Intensity Corrn	Weight %	Atomic %	Element	App. Conc	Intensity Corrn	Weight %	Atomic %
O	71.39	1.1256	33.25	60.30	O	67.75	1.1492	29.80	59
Ca	0.09	1.0367	0.05	0.03	Ca	0.44	1.0445	0.23	0.17
Ni	0.47	0.8558	0.29	0.14	Ni	-	-	-	-
Cr	13.34	0.9904	7.06	3.94	Cr	9.77	1.0102	5.22	2.98
Si	0.16	0.7373	0.12	0.12	Si	0.54	0.7272	0.40	0.42
Cl	24.86	0.8335	15.63	12.80	Cl	21.51	0.8289	10.00	11.72
Fe	74.36	0.8936	43.62	22.66	Fe	80.73	0.9006	54.35	25.70

Table 7: Comparison of EDX analysis of electroplated mild steel samples after pickled with commercial acid and 1N HCl containing 5% PCM.

Element	Electroplated MS samples after pickling with commercial Acid				Electroplated MS samples after pickling with 5% PCM extract				
	App. Conc	Intensity Corrn	Weight %	Atomic %	Element	App. Conc	Intensity Corrn	Weight %	Atomic %
O	25.32	1.0165	11.79	35.19	O	16.51	1.0028	9.53	29.92
Ca	0.10	1.0599	0.04	0.05	Ca	0.09	1.0623	0.05	0.06
Ni	0.54	1.3545	0.19	0.15	Ni	0.25	1.3519	0.11	0.09
Cr	2.74	1.0243	1.26	1.16	Cr	2.55	1.0296	1.43	1.38
Si	-	-	-	-	Si	0.08	0.4874	0.09	0.17
Fe	1.62	1.1277	0.68	0.58	Fe	2.49	1.1310	1.27	1.15
Zn	146.1	0.9630	86.04	62.87	Zn	175.4	0.9684	87.51	67.23

4. Conclusions

The main findings of the above mentioned studies are

1. Peel of *Cucurbita maxima (PCM)* extract act as a good corrosion inhibitor for mild steel in 1N HCl solution and it is a new green inhibitor for metal corrosion inhibition. Strong adsorption of the phytoconstituents of plant extract on the metal surface could be the cause for the inhibitive effect.
2. The phytochemical investigation revealed that the presence of flavonoids, glycosides, proteins, phenolic compounds and tannin in the plant extract.
3. The presence of flavonoid and phenol in the plant extract was also supported by FTIR analysis.
4. The maximum inhibition efficiency of 97% was obtained from the weight loss measurement at 343K.This suggests the PCM protects mild steel from acid corrosion at elevated temperature.
5. Electrochemical measurements confirmed the mixed mode of inhibition. The maximum inhibition efficiency of 85% at 2% v/v concentration of PCM was observed.
6. The adsorption process is spontaneous and follows El-Awady, Temkin and Freundlich adsorption isotherms.
7. The SEM and EDX analysis of commercial acid and PCM extract in industrial pickling process proved that the additions of plant extract minimize the corrosion rate of mild steel considerably. Pickling process with commercial acid, large amount of hydrogen gas evolved but with 5% PCM the hydrogen liberation was very less and controlled. So on a large scale, pickling with1N HCl containing plant extract will be ecofriendly with less fumes and less corrosive effluents.
8. Thus PCM extract proved to be an effective and biodegradable corrosion inhibitor.

5. Acknowledgment

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