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Mild Steel Corrosion Inhibition by *Daucus carota* Pulp Extract in 1N Hydrochloric Acid Medium

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ABSTRACT

Daucus carota pulp extracts (DCP) was investigated as corrosion inhibitor of mild steel in 1N HCl using phytochemical screening, HPLC analysis, conventional weight loss measurement with immersion time, temperature studies, and surface morphological examination. Phytochemical screening and HPLC analysis proved that protective layer was contributed by bioactive constituents DCP extract. Inhibition efficiency increased with increase in concentration of inhibitor from 0.05% to 2.0% v/v. Maximum inhibition efficiency of 95.34% was obtained for DCP extract in 1N HCl for an immersion period of 5h. Scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) techniques were employed for surface examination of uninhibited and inhibited mild steel. These studies provided evidence of improved surface condition on mild steel, due to adsorption of DCP phytonutrients for corrosion protection.

Keywords: *Daucus carota*, Phytochemical Screening, HPLC, Weight Loss Measurements, Surface Examination Studies.

I. INTRODUCTION

Mild steel has good ductility and robustness, high machinability and makes it purpose in different engineering production of some automobile components, its usage in pipelines, buildings, plants, bridges and tin cans [1]. Mild steel are extensively used in chemical and allied industries for handling of acid, alkali and salt solutions [2]. Hydrochloric acid is most complicated acids to handle from standpoints of deterioration and materials of constructions. Hydrochloric acid solutions are widely used in several industrial processes, ore production and oil well acidification. Because of general aggression of acid solutions; inhibitors are commonly used to reduce corrosive attack on metallic materials [3]. Selections of inhibitors are based on its availability, its effectiveness to inhibit substrate material and toxic-free. Excellent inhibitors for corrosion of steel in acidic medium are organic compound containing nitrogen, oxygen and/or sulphur atoms [4-7].

From perspective of safety of metal against corrosion, non-toxic and effectiveness of inhibitors are considered more significant and desirable [8-15]. Aim of this work was to investigate corrosion behaviour and mechanism or mild steel in solutions of 1N HCl with DCP extract by weight loss measurements and surface examination studies.

II. METHODS AND MATERIALS

A. Collection of Plant Material and Preparation of Plant Extracts

Daucus carota plants were collected from local market, Coimbatore. Pulp parts of plant were cut into tiny pieces and shade dried at room temperature for few weeks (Fig. 1) and stored in airtight containers to prevent contamination of material**.** 25g of powdered DCP in 500ml of 1N HCl was refluxed for three hours and kept overnight to extract its phytonutrients (Fig.2). The following day, it was filtered and residue was repeatedly washed with small amount of 1N HCl and filtrate was made up to 500 mL. Prepared extract with 1N HCl was taken as stock solution and required concentrations were prepared by diluting stock solution**.**

Figure 1: Dried pulp materials of *Daucus carota* (DCP)

Figure 2: Photograph of experimental set up for obtaining plant extract using reflux condenser

B. Phytochemical Screening of DCP

Screening of phytochemicals is an important step for detection of bioactive principles present in plants. Phytochemical screening was carried out on DCP extracts by standard procedures [16]. Plant extracts were screened for reducing sugar, alkaloids, protein, phenols, flavonoids, amino acids, tannin, steroids, glycosides and saponins.

C. Analysis of Plant Extract

High Performance Liquid Chromatography (HPLC) analysis was performed for plant extract using HPLC Waters system equipped with 515 HPLC pump and waters 2998 photodiode array detector. Compounds were identified by comparing retention time and reference compounds.

D. Materials Preparation

Corrosion tests were performed on coupons with an active surface of 1cm x 5cm x 2mm cut from sheets of mild steel of 2 mm thickness obtained from Albert Steel House, Coimbatore, India. The chemical composition of mild steel: carbon 0.071%, manganese 0.49%, silicon 0.03%, phosphorus 0.008%, sulphur 0.002%, chromium 0.017%, nickel 0.018% and iron 99.09% was identified at SiTarc, Coimbatore. A hole was drilled at one end of coupons for suspension in the solution. Coupons were degreased with acetone, pickled in conc. HCl and washed with distilled water and coupons were polished with 400, 600 grade of emery papers, cleaned, dried and stored in desiccator to avoid adsorption of moisture [17].

E. Weight Loss Measurements

Weight loss measurements were carried out using a SHIMADZU model AY 220. Mild steel specimens were immersed in beaker containing 100ml acid solution without and with different concentrations of DCP using glass hooks for a predetermined time period at room temperature. For good reproducibility, experiment was carried out in triplicate. Test specimens were removed and washed with de-ionised water, dried and reweighed. Experiments were performed for various parameters such as:

- Concentration variation (0.05%v/v, 0.10%v/v, 0.50%v/v, 1.00%v/v, 1.50%v/v and 2.00%v/v)
- Different time intervals (1h, 3h, 5h, 7h, and 24h)

From the initial and final mass of specimens, weight loss was calculated, and corrosion rate (in mpy) [18, 19] was computed from the following equation:

$$\text{Corrosion rate, CR} = \frac{87.5\ W}{DAT} \quad (1)$$

Where W is the weight loss (mg) of the coupons, D is the density of the coupon (7.8 g/cm^3), A is the surface area of coupon (cm^3), and T is the immersion time (h). Inhibition efficiency of mild steel was then calculated [20].

F. Surface Morphology

Surface of Mild steel coupons were prepared by degreasing with acetone, pickled in conc. HCl and washed with distilled water and coupons were polished with 400, 600 grades of emery papers, cleaned and dried. The coupons were immersed in 1N HCl for determined time period with and without DCP extract. After specified time, the coupons were removed and washed gently with distilled water, dried carefully. Scanning electron microscopy and energy dispersive X-ray techniques were employed for surface examination of uninhibited and inhibited mild steel coupons. SEM provides a pictorial representation on surface to understand nature of the surface film in absence and presence of inhibitors and extent of corrosion of mild steel.

III. RESULTS AND DISCUSSION

A. Phytochemical Analysis

Table 1 shows phytochemical analysis performed on DCP extracts. Result shows that DCP extract was rich in protein, phenol, amino acids and tannins. DCP extract contained oxygen and nitrogen atoms which are centre of inhibitive adsorption metal surface.

The active phyto-constituents in DCP extract may have great influence on their corrosion inhibition property. This suggests that DCP extract can be good and efficient inhibitor of mild steel corrosion within the acid environment.

TABLE 1. Phytochemical Constituents Present in Extract of *Daucus Carota*

S. No.	Phytochemical compound	Result
1.	Reducing sugar	+++
2.	Alkaloids	++
3.	Protein	++
4.	Phenols	++
5.	Flavonoids	+
6.	Amino acids	++
7.	Tannin	+
8.	Steroids	-
9.	Glycosides	+
10.	Saponins	-

Keywords: "++" active compound copiously present, "+" active compound present, "-" active compound absent

B. HPLC Analysis

HPLC analysis was performed to quantify and identify β-carotene in DCP extract. Phenolic compounds were eluted with 89% acetonitrile and 11% dichloromethane as mobile phase at flow rate of 1 mL/min with injection volume 10 µL/min using detection wavelength at 254 nm. DCP extract was quantified and chromatogram of pulp extract was done to determine β-carotene composition in pulp. HPLC fingerprint chromatogram of DCP produced 17 peaks in phenolic compounds (Fig. 3). Peaks of phenolic compounds were identified by comparison of retention times with authentic reference compounds. β-carotene appeared at 3.653 min in chromatogram. Phenolic compounds contribute for inhibition efficiency of DCP extract. Composition of β-carotene was quantified in DCP extract and was found to be 2.47 mg/100 g.

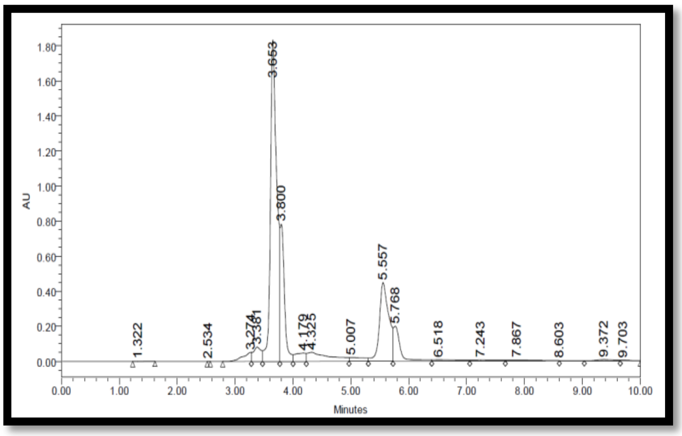

Figure 3: HPLC chromatogram of DCP extract

C. Weight Loss Measurements

Table 2 gives values of inhibition efficiency obtained from weight loss measurements of mild steel for various concentrations of DCP in 1N HCl at 303K after different hours of immersion [21]. Inhibition efficiency was calculated using the formula

$$IE\% = \frac{W_1 - W_2}{W_1} \times 100 \qquad (2)$$

Where, W_1 and W_2 are weight loss of mild steel after immersion in solutions without and with inhibitor respectively.

As the concentration of inhibitor increases, rate of corrosion decreases because inhibitor molecules prevent dissolution of mild steel by effective adsorption of phytonutrients of plant extract on metal surface area (Fig. 4). Adsorbed organic molecules prevent further interaction of metal with acid.

TABLE 2.
Inhibition Efficiency (IE) of DCP/1N HCl against Mild Steel at various Concentrations and different Immersion Period

Conc. of extract in v/v %	Inhibition Efficiency (IE %)				
	Immersion time in hours				
	1	3	5	7	24
0.05	67.35	69.13	74.53	72.52	70.86
0.10	73.47	74.50	75.47	73.18	72.84
0.50	77.55	79.87	85.71	80.79	79.10
1.00	78.57	80.54	86.34	83.77	81.71
1.50	84.69	85.91	89.44	87.09	84.64
2.00	86.73	87.25	95.34	92.05	88.28

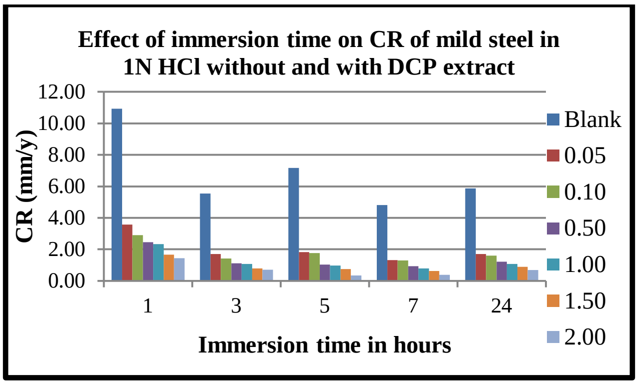

Figure 4: Effect of immersion time on CR of mild steel in 1N HCl without and with DCP extract

1) Effect of concentration of DCP on Corrosion rate and Inhibition Efficiency

Variations of inhibition efficiency and corrosion rate with change in concentration of DCP/1N HCl are presented in Table 2. From the data it was obvious that there was decrease in corrosion rate with increase in inhibitor concentration for all immersion periods. Decrease in corrosion rate and increase in inhibitor efficiency was usually attributed to adsorption of plant constituents on surface of mild steel which makes a barrier for mass and charge transfers and protects further attack by acid [14]. Plant phytoconstituents are molecules capable of covering a large surface area on adsorption. Molecules such as tannins and flavonoids are responsible for high inhibition efficiency at relatively low concentration of inhibitor. This accounts for no further increase in inhibition efficiency with increase in concentration greater than 2.0% [15]. This finding shows that inhibitor has capability of protecting surface by forming a passive film.

2) Effect of Immersion Time on Corrosion Rate and Inhibition Efficiency

Inhibition efficiency increased with increase in concentration of inhibitor from 0.05% v/v to 2.0% v/v at room temperature. Maximum inhibition efficiency of 95.34% was found for immersion period of 5h in DCP/1N HCl. Decrease in inhibition efficiency thereafter with increasing time may be due to shift in adsorption and desorption equilibrium which takes place simultaneously on prolonged exposure to corrosive media [22]. These results suggest that adsorption model arrangement and orientation of constituents present in DCP extract on surface of mild steel may change with time [23]. Therefore for higher concentration of inhibitor, more number of inhibitor molecules gets absorbed on surface of mild steel [24].

D. Surface Examination Studies

Corrosion inhibition efficiency of DCP in 1N HCl on mild steel was supported by the Scanning electron microscopy (SEM), X–ray Diffraction **(**XRD), Energy dispersive X-ray (EDX).

1) Scanning Electron Microscopy (SEM) Analysis

Surface morphological characteristics of uninhibited mild steel in 1N HCl and inhibited mild steel using DCP in 1N HCl were analysed at an accelerating voltage using JOEL SEM model JSM 6390. In the SEM photograph of polished mild steel (Fig. 5), no pits or cracks were observed except polishing lines [25]. Inspection of Fig. 6 revealed that specimens immersed were rough and extremely damaged in presence of 1N HCl [24]. Depth of surface corroded due to exposure of mild steel to acid was well visible with shallow pits, pores and cracks [26].

SEM photograph (Fig. 7) of mild steel exposed to acid containing inhibitors shows that there was less damage on the mild steel surface which clearly confirms inhibitive action due to formation of protective film by phytochemical components present in DCP extract on mild steel surface resulting in decrease in contact between metal and aggressive medium and effectively exhibit inhibition effect [27].

SEM photographs of mild steel samples

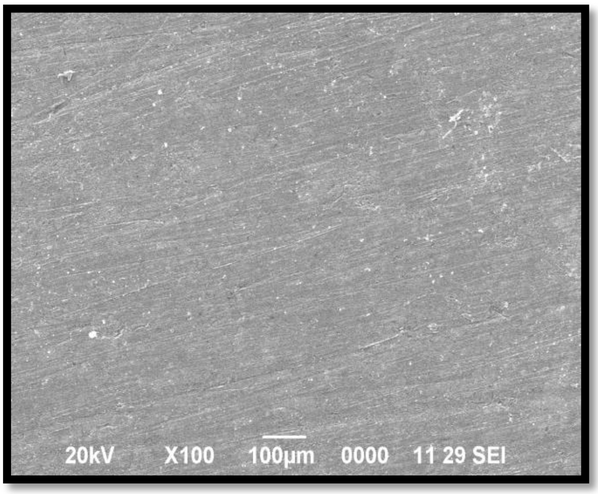

Figure 5: Polished mild steel surface

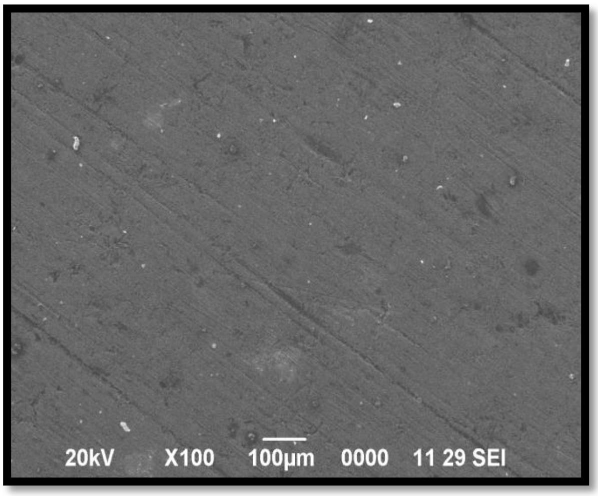

Figure 6: Mild steel surface exposed to 1N HCl for 5h at 303K

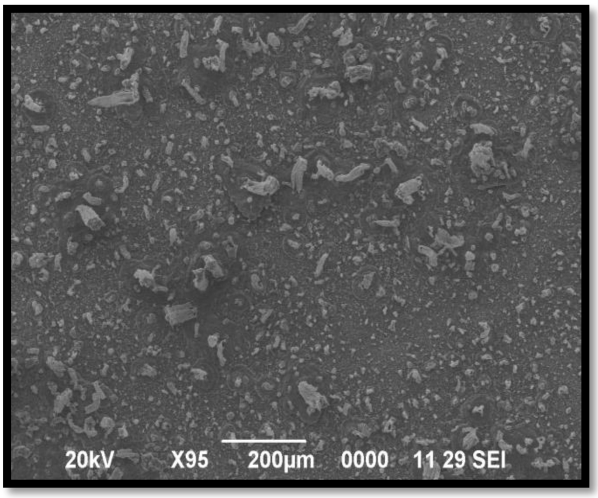

Figure 7: Mild steel surface exposed to 1N HCl + 2% DCP for 5h at 303K

2) X–ray Diffraction (XRD) Studies

X-ray diffraction was used to determine film formation on mild steel in various test solutions. Corrosion product over the surface of mild steel in 1N HCl solution was shown in Fig. 8. Peaks at 2θ = 44, 82 and 65 can be assigned to oxides of iron (Fig.9).

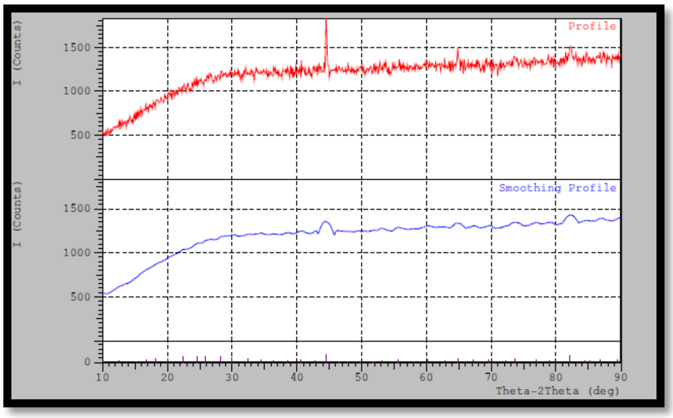

Figure 8: XRD spectrum of mild steel corrosion in absence of DCP extract.

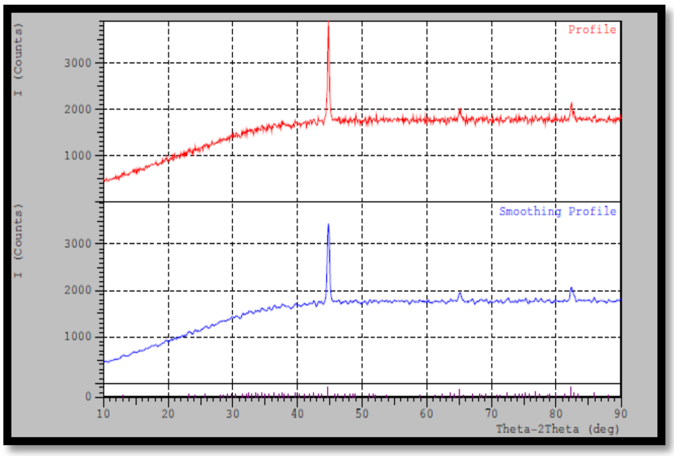

Figure 9: XRD spectrum of mild steel corrosion in DCP in 1NHCl.

It can be observed that in absence of inhibitor, surface of metal contains oxides of iron. XRD patterns of inhibited surface (Fig. 9) showed presence of iron peaks only, intensity of peaks due to oxides of iron was found to be absent [28]. Formation of adsorbed protective film on surface of metal in presence of DCP extract was clearly reflected from these observations.

3) Energy Dispersive X–ray Spectroscopy (EDX) Studies

EDX spectra employed to examine composition of corrosion scales on mild steel immersed for five hours in

1N HCl without and with inhibitor containing 2% v/v concentration of DCP extract are shown in Fig. 10 and 11. EDX results summarized in Tables 6 - 8, indicates presence of silicon, potassium, oxygen and chlorine elements on mild steel surface exposed to DCP /1N HCl, in addition to iron and carbon in mild steel. Atom percentage of elements carbon, silicon, oxygen, chlorine and potassium suggest adsorption of nutrients present in extract on mild steel surface enhancing inhibition properties through complex formation with metal atoms [29].

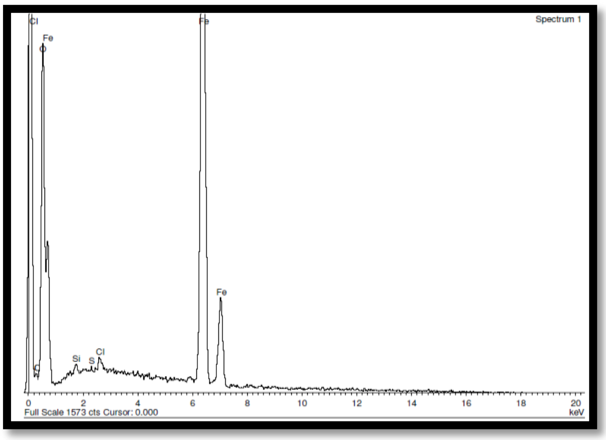

Figure 10: EDX spectrum of mild steel in 1N HCl

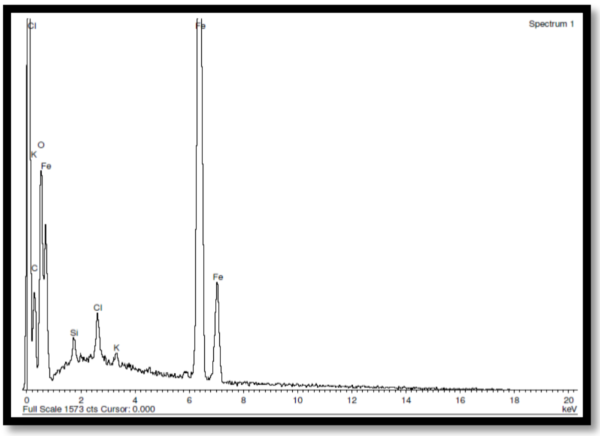

Figure 11: EDX spectrum of mild steel in DCP/1N HCl

TABLE 6. EDX analysis result of mild steel immersed in DCP in 1N HCl extract

Sample	Element Weight %				
	C	**Fe**	**Si**	**Cl**	K
Mild steel exposed to HCl	-	70.17	0.45	0.53	-
Mild steel exposed to DCP	30.70	46.55	0.63	1.10	0.31

TABLE 7. EDX analysis result of mild steel immersed in 1N HCl

Element	**Weight %**	**Atom %**
O	28.78	58.24
Si	0.45	0.52
S	0.07	0.07
Cl	0.53	0.48
Fe	70.17	40.69
Total	100	100

TABLE 8. EDX analysis result of mild steel immersed in DCP/1N HCl

Element	**Weight %**	**Atom %**
C	30.70	53.86
O	20.72	27.29
Si	0.63	0.47
Cl	1.10	0.65
K	0.31	0.17
Fe	46.55	17.56
Total	100.00	100.00

EDX micrograph of mild steel surface after immersion for 5h in 2%v/v concentration DCP extract are shown in Fig. 12 and Fig. 13. Micrograph of mild steel exposed to acid containing inhibitors shows the presence of adsorbed inhibitor molecules on surface which prevented the corrosion [30].

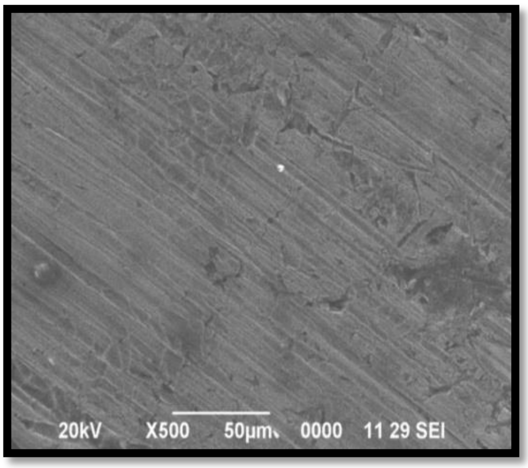

Figure 12: EDX micrograph of mild steel exposed to 1N HCl

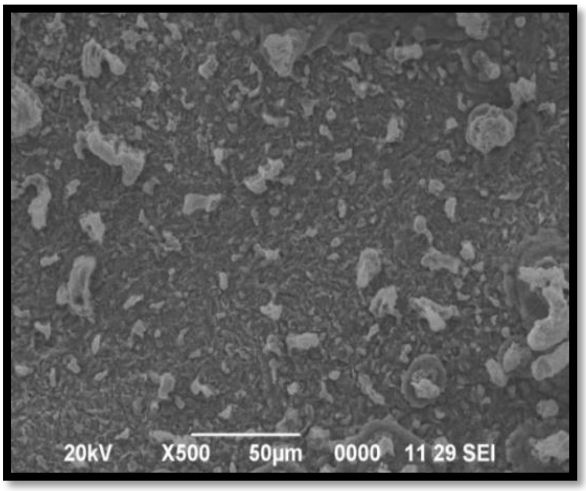

Figure 13: EDX micrograph of mild steel exposed to DCP/1N HCl

IV. CONCLUSION

Based on the above results, following conclusion can be drawn

- DCP extract of plant was found to be an effective inhibitor for corrosion of mild steel in 1N HCl medium. Inhibition efficiency increased with an increase in DCP extract content.
- Weight loss measurements, phytochemical screening and surface examination studies confirmed the inhibitive nature of the DCP in 1N HCl medium.
- Phytochemical screening proved that DCP extract was rich in protein, phenol, amino acids and tannins which contribute to inhibitive property of the extract.
- Composition of β-carotene in DCP was 2.47 mg/100g reported using HPLC analysis.
- Inhibition efficiency increased with increase in concentration of inhibitor from 0.05% v/v to 2.0% v/v at room temperature. Maximum inhibition efficiency was 95.34% in DCP/1N HCl for immersion period of 5h.
- SEM photographs of mild steel exposed to acid containing inhibitors confirm inhibitive action due to formation of protective film by phytochemical components of DCP extract.
- XRD study confirmed blockage of metal surface through adsorption process.
- EDX studies indicates presence of silicon, potassium, oxygen and chlorine elements on mild steel surface exposed to DCP in 1N HCl that confirms presence of adsorbed inhibitor molecules on mild steel surface.

DCP extract was thus proved to be an effective green corrosion inhibitor with cost effective to be commercially applied in industries.

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