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Anti- corrosion behaviour of *Hydnocarpus pentandra* on mild steel in acid medium

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Abstract
Corrosion is the degradation of the metal surface by the reaction with its environment. In today's circumstance corrosion is one of the major problem in the industry and factories and other manufacturing sectors. The present work investigates corrosion inhibition of mild steel in 1N HCl in presence of *Hydnocarpus pentandra* seed (HPS) extract which is eco- friendly, biodegradable and does not pollute the environment. Corrosion inhibition properties of (HPS) extract has been examined on mild steel corrosion in 1N HCl solution by using Phytochemical analysis, weight loss method, FT-IR analysis, SEM and EDAX analysis. The result reveals that an increase in concentration of inhibitor, decrease the corrosion rate and rate of degradation of the metal. The best inhibition effect of HPS extract for mild steel was obtained by the weight loss measurements up to 97%. Due to existing of various functional groups and phytochemical constituents the inhibition efficiency increases and with increase in the concentration of HPS extract. The surface morphology of inhibited and uninhibited mild steel was investigated by scanning electron microscope. The elemental composition was studied by the energy dispersive x-ray analayis.

Keywords: *Hydnocarpus pentandra*, phytochemical analysis, weight loss method, FT-IR Analysis, SEM – EDAX Analysis

Introduction
Corrosion is a destruction of metal surface by the surrounding environment. Like other natural hazards, corrosion can cause dangerous and costly damage to everything from vehicles, equipment, water supply, drainage systems to pipes, bridges and public buildings. Mild steel is a low carbon steel, widely used forms of steel and can be applied to other applications. Mostly Mild steel are low cost and easily available. Mild steel exhibit properties like excellent machinability and weldability. So protection of mild steel is an essential in the industry and other fields from corrosion. Corrosion can prevented by various methods like protecting coatings, dyes, anodic and cathodic protection and inhibitors are also used. The inhibitors are classified into natural and synthetic depending on the raw materials we use. In recent times chemicals used in industry and factories causes various pollution to the environment. So eco-friendly and bio-gradable green corrosion inhibitor is synthesized and used for corrosion inhibition for acid medium. The seeds of *Hydnocarpus pentandra* collected from the Anaimalai region, Pollachi. From the seed, extract is prepared by 1N HCl, which is used for corrosion inhibitor. In this study a eco – friendly, natural inhibitor is synthesised from the *Hydnocarpus pentandra* seeds in 1N HCl medium used as a corrosion inhibitor for the acid medium. The corrosion study involves phytohemical study,weight loss method, FT-IR, SEM –EDAX analyses.

Materials and methods
1. Preparation of mild steel
The sheet of mild steel used for this study has 2 mm in thickness and was mechanically press cut into 5cm x 2cm coupons.

2. Preparation of plant extract
The seeds of *Hydnocarpus pentandra* was collected from the locations in Anaimalai region at Pollachi. The seeds of *Hydnocarpus Pentandra* was dried and grounded. The HPS extract of 5 % to be prepared 0.50g of grounded seeds was taken in the round- bottom flask with 1 L of 1N HCL acid. The RB flask was fixed with reflux condenser and subjected for 3 hours of heat. The refluxed solution was allowed to stand for overnight, filtered and stored. The stock solution was used for the further analysis.

3. Phytochemical Analysis
Phytochemical analysis of the HPS extract of the samples were carried out in different test tubes. The procedure was discussed below;
Test for tannins -To the HPS extract, add 2 ml of lead acetate, formation of yellow precipitates indicates the presence of tannins. Test for phenol -To the HPS extract, add 1 ml of neutral $FeCl_3$, formation of green colour indicates the presence of phenol group. Test for coumarin- To the HPS extract add 1ml of NaOH or aq.NH_3, formation of yellow colour indicates the presence of Coumarin. Test for anthocyanin - To the HPS extract, add few ml conc. H_2SO_4, formation of orange colour indicates the presence of anthocyanin group. Test for glycosides -To the HPS extract was dissolved in 2 ml of water and then add aq. NH_3 or NaOH., formation of yellow colour indicates the presence of glycosides. Test for saponins-To the HPS extract, add 6 ml of distilled water and shaken for few minutes, formation of foam indicates the presence of saponins. Test for flavones - To the HPS extract, add 2ml of 10% NaOH., formation of yellow to brown colour indicates the presence of flavones. Test for quinines - To the HPS extract, add few ml of dil. NaOH, formation red colour indicates the presence of quinone group. Test for carbohydrates - To the HPS extract, add 2 ml of alcohol with α– Napthal crystal, add conc. H_2SO_4, shaken well, formation of reddish – violet colour ring at the junction, indicates the presence of Carbohydrates group. Test for proteins -To the HPS extract, add 40% of NaOH and 1 ml Copper Sulphate, formation violet colour indicates the presence of protein group. Test for

antraquinones -To the HPS extract, add few ml of benzene and add 10 % ammonia, formation pink or red colour indicates the presence of antraquinone.

4. Weight Loss Determination

The weight loss method was used to optimize the concentration of the inhibitors. Initially the mild steel specimen was weighed and noted in the table. The mild steel were immersed in 100ml of inhibited and uninhibited solutions for various Time-intervals at room temperature. After the immersion the specimens were withdrawn, rinsed with distilled water, dried in the desiccators and weighed accurately. Weight loss measurements were also performed at various immersion time like 1hour, 3 hours, 5 hours, 7 hours and 24 hours for the different concentration of the HPS extract at room temperature. From the table, corrosion rate (mpy) and the inhibition efficiency were calculated from the table by the following equations:

$$\textbf{Corrosion rate} = (534\ x\ W)\ /\ \textbf{DAT}$$

Where,
W- Weight loss of the mild steel
D - Density of mild steel (kg /m^3)
T - Time of immersion
A - Area of the specimen exposed to the corrosive solution (m^2)

$$\textbf{Inhibition Efficiency} = \left(\frac{\textbf{CR (blank)} - \textbf{CRi}}{\textbf{CR (blank)}}\right) \textbf{x } 100\%$$

Where,
CR (blank)-Corrosion rate of the blank solution (mpy)
CRi - Corrosion rate of the inhibitor(mpy)

5. FT-IR Analysis

FT-IR determine the localization area and characterization of corrosion in the fingerprint region. FT-IR spectra gives presence of various functional groups present in the mild steel specimen, when it reacted with the HPS extract. Mild steel in presence and absence of optimum concentration of HPS extract after an immersion time of 3 hours mild – steel specimen samples. After completion of the experiment, the specimens were removed and dried with the drier and in the desiccator. Three different samples were taken with different concentrations like blank, 1 % and 3 % with HPS extract.

6. SEM – EDAX Analysis

The analysis of morphological character of the mild steel was studied by the SEM analysis and determination of the elemental composition of the mild steel was studied by the EDAX analysis. The scanning electron microscopic micrograph of mild steel in the presence and absence of optimum concentration of HPS extract after an immersion time of 3 hours. After completion of the experiment, the specimens were removed and dried with the drier. Three different samples were taken with different concentrations like blank,1 % and 3 % with HPS extract.

Results and discusstions

1. Phytochemical screening

The phytochemical constituents of HCl extract of *Hynocarpus pentandra* extract. The constituents analysed was noted in the table.1. The result indicates that the inhibitive and adsorption properties of HCl extract of *Hydnocarpus pentandra* is due to the presence of Tannins, phenol, saponins, flavones, carbohydrates and glycosides in the extract of seed.

Phytochemical Constituents	Observation
Tannins	+++
Phenol	+++
Coumarin	-----
Anthocyanin	-----
Glycosides	+++
Saponins	+++
Flavones	+++
Quinones	-----
Carbohydrate	+++
Proteins	-----
Antraquinones	-----

2. Weight loss method

The corrosion parameters like inhibition efficiency (IE) and corrosion rate (CR) at various concentrations like blank,0.01,0.05,0.1,0.5,1,1.5,2,2.5 and 3 of inhibitors was determined and presented in the table: 1. The results shows that the corrosion rate of the mild steel in 1N HCl decreases with increase in the concentration of the HPS extract is shown in the Fig **1**. This suggests that the concentration of the extract increases, destruction of mild steel reduces gradually. The inhibition efficiency of mild steel exposed to different concentrations of *Hynocarpus pentandra* extract in 1N HCl at room temperature is shown in the Fig.2**.** The inhibition efficiency increases gradually, when the concentration of HPS extracts increases. The maximum percentage inhibition of 97% was recorded at the highest concentration studied at the room temperature by treating with HPS extract.

Table 1: Weight loss method of HPS in 1N HCl at different immersion time

Conc. of Extract	1 hour		3 hour		5 hour		7 hour		24 hour	
	CR (mpy)	IE %	CR (mpy)	IE %	CR (mpy)	IE %	CR (mpy)	IE %	CR (mpy)	IE %
Blank	785	-	640	-	270	-	343	-	142	-
0.01	349	56	189	70	87	68	100	71	45	65
0.05	349	56	174	73	87	68	87	75	49	68
0.1	305	61	116	82	78	71	69	80	36	74
0.5	262	67	116	82	70	74	56	84	24	83
1	262	67	101	84	70	74	50	85	22	85
1.5	218	72	87	86	61	77	50	85	13	91
2	218	72	73	89	52	81	44	87	9	94
2.5	174	78	72	88	52	81	37	89	5	96
3	87	88	58	91	35	87	31	91	4	97

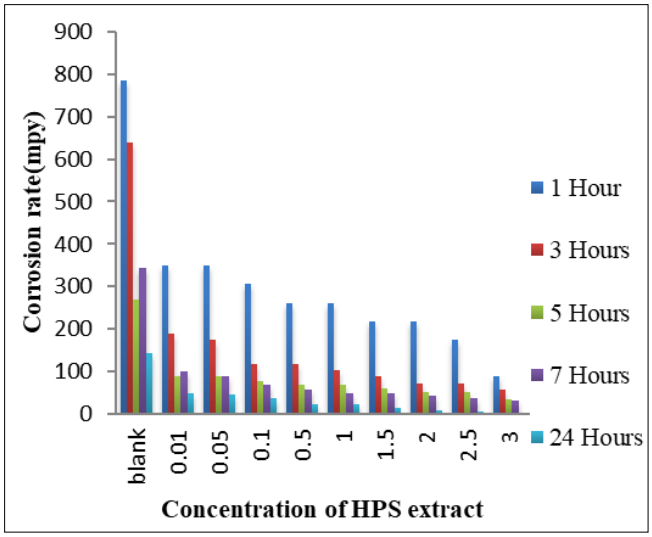

Fig 1

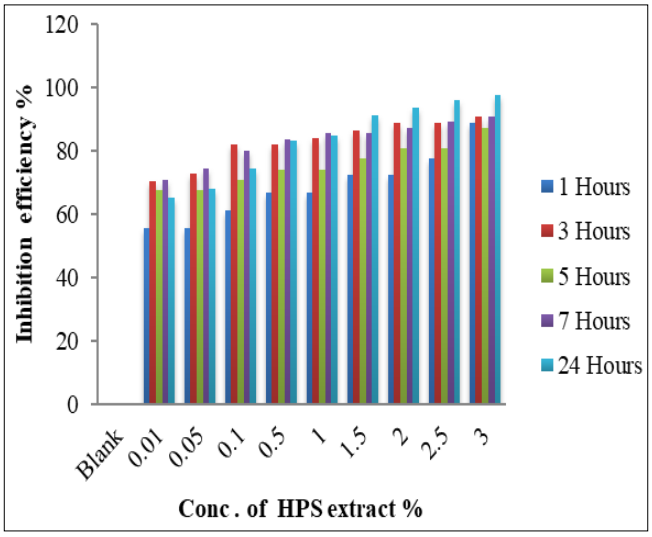

Fig 2

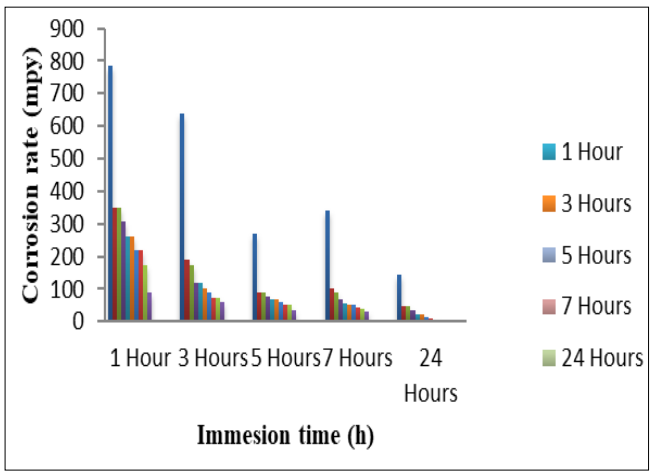

Fig 3

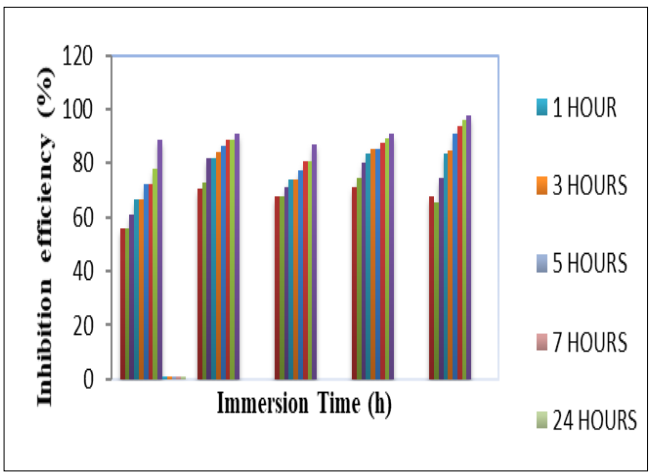

Fig 4

3. FT-IR Analysis

The FT-IR provides us with the basic idea about the functional groups present in the inhibitor molecule. The Fig: 6 shows that FT-IR spectrum of the seed extract of *Hynocarpus pentandra*. The Fig: 7 shows that FT-IR spectrum of mild steel at the blank. In the blank there is the absence of HPS molecules on the specimen. The HPS extract adsorbed on the surface of mild steel were studied to evaluate the main functional groups. The spectrum of inhibitor adsorbed on the mild steel surface shows almost all the characteristic peaks.IR spectra of the specimen mild steel with 1% HPS and 3 % extract showed in the fig. 8 and 9 shows the presence of groups like OH, C=O, O = C=O and etc. This shows the anticorrosive properties of the mild steel by the HPS extract reacted with the mild steel.

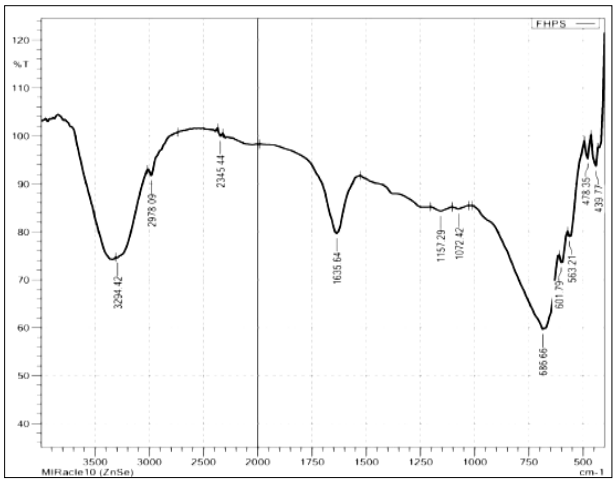

Fig 6

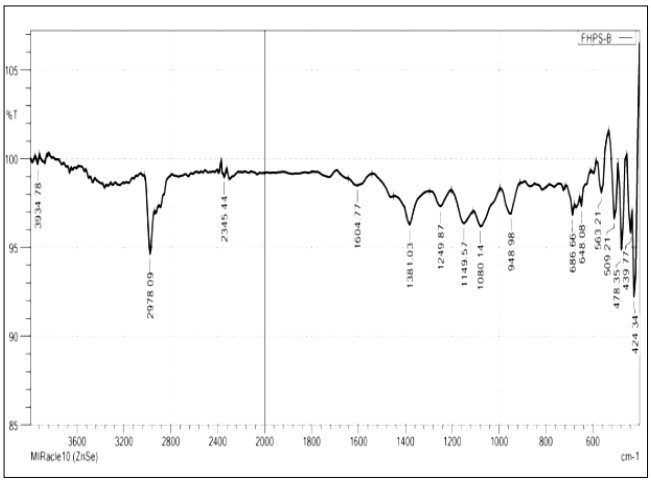

Fig 7

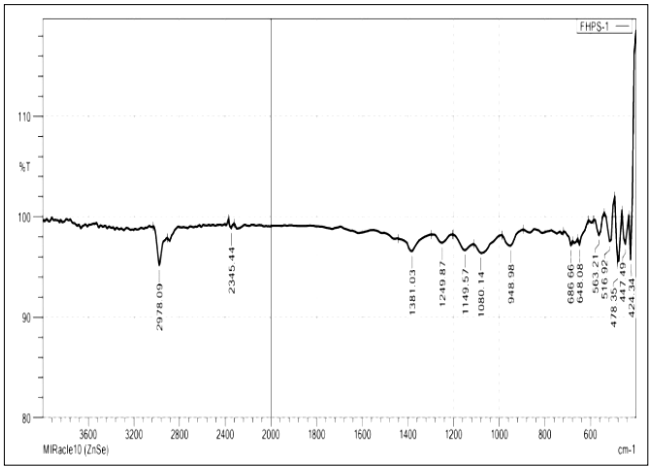

Fig 8

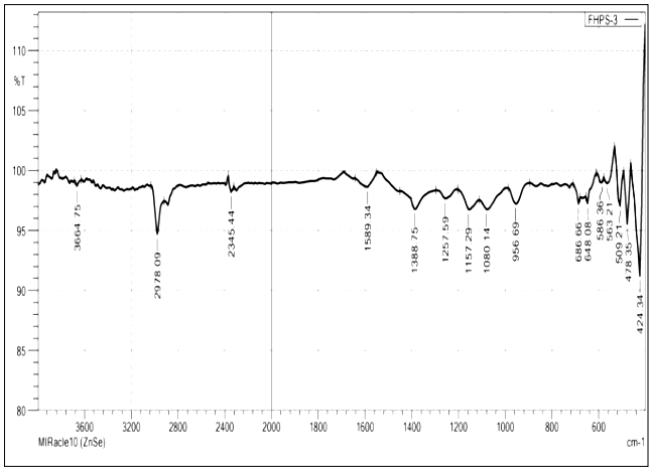

Fig 9

Table 3: FTIR peak values of Table

FT-IR peak values	
Crude HPS	**Possible functional Group**
3294.42	O-H stretch
2978.09	C-H stretch
1635.64	C =O stretch
1157.29	C-O stretch
686.66	C=C bending
563.21	C-X stretch

Table 4: FTIR peak values of mild steel in crude HPS extract. blank and HPS extract.

FT-IR peak values			
Mild steel In blank	**Mild steel In 1%HPS**	**Mild steel in 3%HPS**	**Possible Functional Group**
-	-	3664.75	O-H stretch
-	2978.09	2978.09	C-H stretch
-	1388.03	1388.75	C-H bending
-	-	1157.29	C-O
686.66	-	-	Fe_2O_3
-	648.08	648.08	C=C
563.21	-	-	FeO
-	-	586	C-X stretch

4. SEM Analysis (Scanning Electron Microscope)

Surface analysis of the mild steel was made using scanning electron microscope with the different magnificent. It is clear that the surface of mild steel was strongly damaged in the HCl solution without HPS Extract. (i.e.) it has rough surface. The Fig.10. shows a dissolution of the mild steel in exposure with HCl solution without inhibitor. The visual performance of the mild steel surface exposed to HCl solution was significantly changed in the presence of HPS extract.

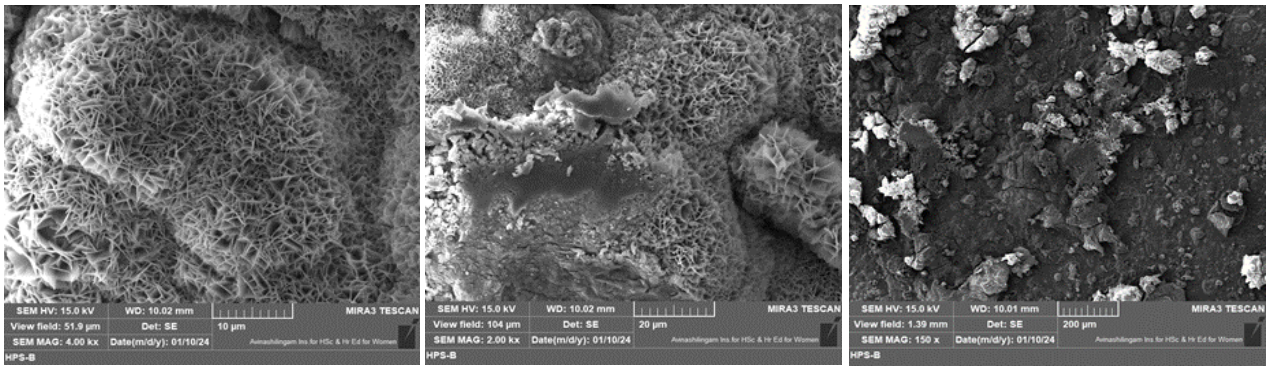

Fig 10: SEM images of mild steel in 1N HCl

It can be seen from the Figs.11 and 12 below. That the HPS extract reduced destruction rate of mild steel and lower corrosion products were formed on the mild steel surface in the presence of inhibitor. The lowest surface damage were observed at the highest HPS extract (Fig 11). The SEM studies showed that the inhibited mild steel surface was found smoother than the uninhibited surface due to the formation of protective film on the inhibited mild steel surface.

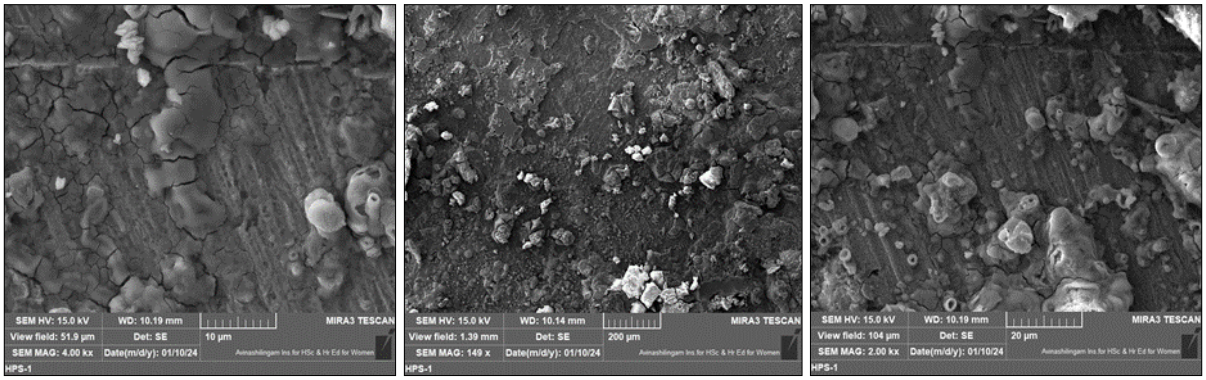

Fig 11: SEM images of the mild steel in 1% HPS.

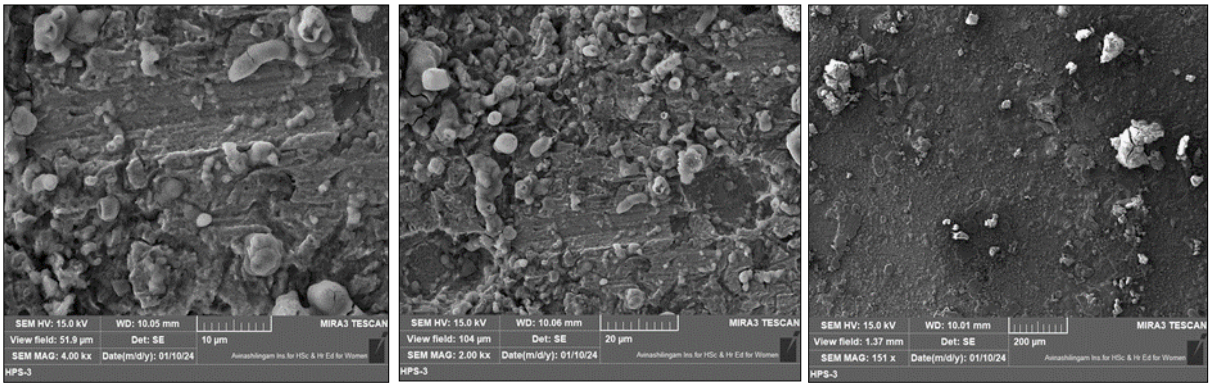

Fig 12: SEM images of the mild steel in 3 % HPS

5. EDAX Analysis (Energy Dispersive X-ray)

EDAX Analysis is used to determine the elemental composition of the mild steel in presence and absence of HPS extract. Fig 12, 13 and 14 represents the EDAX analysis of mild steel in the absence and presence of an optimum concentration of HPS extract. Fig:12 Depicted the EDAX analysis of mild steel in the absence of HPS extract. It was observed that the EDAX analysis of mild steel in the absence of HPS extract, shows signals for carbon (2.86%), oxygen (26.74%) and iron (70.41%) elements. The EDAX spectra of mild steel in the presence of 1% HPS extract shows the characteristic signals for carbon (3.67%), oxygen (25.49%) and iron (70.84%), this shows in the fig 13

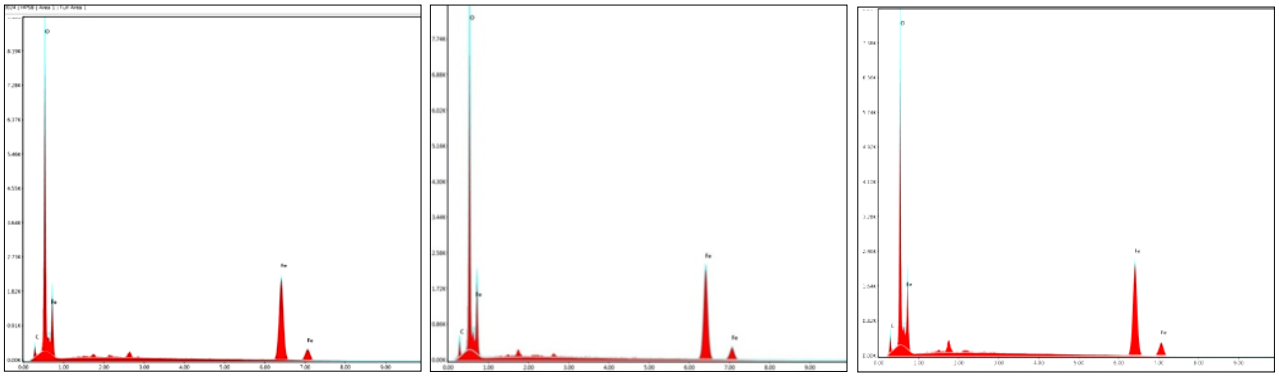

Fig 12 **Fig 13** **Fig 14**

Table 2: EDAX analysis of mild steel

Concentration	Element	Weight %	Atomic %
Blank	C K	2.86	7.51
	O K	26.74	52.72
	Fe K	70.41	39.77
1%	C K	3.67	9.65
	O K	25.49	50.30
	Fe K	70.84	40.05
3%	C K	4.23	11.14
	O K	24.47	48.43
	Fe K	71.30	40.42

Conclusion

From the results and findings of the study, the following conclusions are made

- The *Hydnocarpus pentandra* seed extract is a good eco-friendly green inhibitor for the mild steel in 1N HCl solution.
- The inhibition efficiency of the extract is due to the presence of saponins, tannins, phenol, glycosides, flavones and carbohydrate.
- From the weight loss method, Inhibition efficiency of HPS extract increases up to 97% at higher concentration.
- Corrosion rate of the mild steel decreases with increased concentration of HPS extract.
- Inhibition efficiency of HPS extract increases with increased concentration of HPS extract and inhibit the corrosion of mild steel in acid medium.
- The seed extract of *Hydnocarpus pentandra* contains various functional groups confirmed by FT-IR analysis.
- SEM and EDX analysis showed that the inhibited mild steel surface was found smother than the uninhibited surface due to the formation of protective film on the mild steel surface.
- The above results proved that *Hydnocarpus pentandra* seed extract is a natural corrosion inhibitor, successfully applied as a corrosion inhibitor on the mild steel in the acidic medium.

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