A STUDY ON RESISTIVITY POWER OF FLUEGGEA LEUCOPYRUS ON CARBON STEEL AGAINST CORROSION

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ABSTRACT

The ethanolic extract of *Flueggea leucopyrus* was used as corrosion inhibitor on carbon steel in acidic medium. The phyto constituents, antimicrobial activity and GC-MS analyses were carried out for the plant extract. The resistivity power of *Flueggea leucopyrus* was determined by weight loss method, for various concentrations of the extract with Zn^{2+} ion and *tartaric acid, citric acid* and *lactic acid* were used as additives in 0.5M HCl medium. The resistive film that protects the surface of the metal was confirmed by the spectral studies such as FTIR, UV-Visible and fluorescence spectra. The film formation is also confirmed by the surface morphology studies such as SEM and EDAX analyses. It also shows the formation of complex between the metal cation, additives and the compounds present in the extract of *Flueggea leucopyrus.*

INDEX TERMS: Anticorrosion, Resistivity power, *Flueggea leucopyrus*, GC-MS, EDAX, SEM

I. INTRODUCTION

Now-a-days metal are used almost in all fields such as industries, power stations and home appliances. Metallic corrosion is the process of deterioration on the metal surface through the interaction with the moisture in the environment. Corrosion is a natural deterioration process which can be controlled but cannot be completely prevented. In past years, chemical inhibitors were used to control corrosion which was found to be hazardous and toxic later. So eco-friendly, non–toxic chemical inhibitors were suggested and reported. In recent days, green inhibitors from natural products have been used as inhibitors which are eco-friendly and completely non-toxic[1-3]. *Flueggea leucopyrus* is a plant which belongs to the family '*Euphorbiaceae'*. It is a bushy shrub. It is used as a disinfectant and for treating seminal weakness and burning sensations[4]. *Flueggea leucopyrus Willd* which are found in Pachmarhi (Dhupgarh) consists of 28 chromosomes and 4x ploidy level[5].The plant has been used as disinfectant, fish poison. It is also used as medicine for cough, asthma, diarrhea, mental disorders and kidney stone[6-7]. The quantitative analysis of the plant shows that it has good medicinal properties especially anti-cancer property which is justified by the presence of phenolics, flavanoids and saponins in large percentage[8]. The decoction of the plant is very effective in the treatment of breast cancer[9]. The plant also acts an antioxidant and it induces apoptosis[10].

The present determination is done,

- To analyse the phytoconstituents, antimicrobial activity and GC-MS for the ethanol extracted leaves of *Flueggea leucopyrus.*
- To evaluate the resistivity power of *Flueggea leucopyrus* (FL) by the inhibition efficiencies of FL-Zn^{2+}, FL-Zn^{2+}-Tartaric acid, FL-Zn^{2+}-Citric acid, FL-Zn^{2+}-Lactic acid systems in resisting the corrosion on carbon steel in acidic medium.
- To analyze the composition of the film formed on the metal surface by the spectral studies such as
  - FT-IR
  - UV-Visible
  - Fluorescence
- To analyze the surface morphology of the film formed on the surface of the metal by
  - Scanning Electron Microscope (SEM)
  - Energy Dispersive X-Ray Analysis (EDAX)

Fig.1: *Flueggea leucopyrus*

II. MATERIALS AND METHODS

2.1 Metal Specimens

The metal specimens taken for this study is carbon steel with the composition (wt %) of S-0.026, P-0.06, Mn-0.4, C-0.1 and balance iron. The dimensions of the metal active surface are 1.2 X 4.1 X 0.2 cm which was used for weight loss measurements. The carbon steel specimens were mechanically polished, washed in double distilled water and degreased with acetone and used for the weight loss method and surface examination studies.

2.2 Extraction

The leaves of *Flueggea Leucopyrus* were collected from "*Pachaimalai*" hills near Trichy district located at 119^0N, $78^0$21'E of Trichy. The leaves were washed thoroughly several times in the running tap water and it was taken and dried under shade. About 100g of the powder was soaked in 500ml of ethanol under cold percolation method. At regular intervals of time the extract was filtered and distillation was carried out to collect the crude extract [11] and taken for preliminary qualitative phytochemical screening analysis[12] and for further studies.

2.3 GC-MS Analysis

GC-MS plays a key role in the analysis of unknown components of plant origin. GC-MS ionizes compounds and measures their mass numbers. This technique involves the separation of volatile components in a test sample using suitable capillary column coated with polar and non-polar or intermediate polar chemicals.

2.4 Weight-Loss Method

2.4.1 Determination of Corrosion Rate

Weight loss measurements were carried out using an ACCULAB Electronic top loading balance, with accuracy. The specimens were immersed in the beaker containing 100ml of acid solutions without and with different concentration of *Flueggea Leucopyrus* leaves extract using hooks. Before it was immersed, the specimens were cleaned and the weight is recorded. After 72 hours, the test specimens were removed and then washed with double distilled water, dried and reweighed. The average mass loss of two parallel carbon steel specimens was obtained.[13] From the difference in weight of specimens the corrosion rate was calculated by the following expression,

$$\text{Corrosion Rate} = \frac{87.6 \times W}{A \times T \times D} \quad \text{(mpy)}$$

W = Loss in weight in mg, A = Surface area of the specimen (cm^2), T = Time in hours (h),

D = Density (7.2 g/cm^3)

Corrosion Inhibition Efficiency (IE) was then calculated using the equation

$$IE = 100[1-(W_2/W_1)]\ \%$$

Where,

W_1 = Corrosion rate in the absence of inhibitor and

W_2 = Corrosion rate in the presence of inhibitor

2.5 Infrared (IR) Spectroscopy

Infrared spectroscopy is a well developed technique to identify the functional groups of the chemical compounds. The specimens were suspended by means of hooks in solution having with and without inhibitor for 72 hours. After 72 hours the specimen were taken out. Then the film formed on the metal surface was scratched off and taken for FTIR spectral study.

2.6 UV-Visible Spectroscopy

The possibility of the formation of film on the metal surface was examined by mixing the respective solution and recording their UV-Visible absorption spectra using Lambda 35 UV-Visible spectrophotometer which is a PC controlled single beam scanning spectrophotometer. It covers wavelength range from 200 nm to 1000 nm with a setting accuracy of ± 1 nm.

Phyto-constituents	Inferrence	Phyto-constituents	Inferrence
Carbohydrates	+	Anthraquinone Glycosides	+
Reducing Sugar	+	Saponin Glycosides	+
Hexose Sugar	+	Cyanogenic Glycosides	-
Non-Reducing Sugar (Starch)	+	Alkaloids	+
Proteins	+	Tannins	+
Amino Acids	+	Phenolic Compounds	+
Tyrosine	+	Flavonoids	+
Steroids	+	Terpenoids	+

2.7Fluorescence Spectroscopy

Fluorescence spectra of the film formed on the metal surface were recorded using Jasco F-6300 Spectrofluorometer.

2.8 SEM Analysis

A Scanning electron microscope (SEM) is a type of electron microscope that images a sample by scanning it with a beam of electrons in a raster scan pattern. The specimens were suspended by means of hooks in solution having with and without inhibitor for 72 h. Then the specimens were taken out and the metal specimens were examined.

2.9 Energy Dispersive Analysis of X-Rays (EDAX)

The carbon steel immersed in blank solution and in the inhibitor solution for a period of 72 h were removed, rinsed with double distilled water, dried and observed in an Energy Dispersive Analysis of X-Rays (EDAX) to examine the elements present on the carbon steel surface. The elemental analysis of the carbon steel surface was carried out using an energy dispersive X-ray analyzer (EDAX) unit attached to the SUPRA 55 Field Emission Scanning Electron Microscope (FESEM).

III. RESULTS AND DISCUSSION

3.1 Qualitative preliminary phytochemical screening

Table (1) illustrates that the active phyto compounds such as alkaloids, terpinoids, flavanoids, phenolic compounds, proteins etc. are present except cyanogenic glycosides. The presence of these active compounds is responsible for the inhibitive nature of the plant extract.

Table (1): Qualitative preliminary phytochemical screening of ethanolic extract of *Flueggea Leucopyrus Willd* (FL)

Glycosides	+	Saponins	+

3.2 Antimicrobial studies

3.2.1 Antibacterial activity

The evaluation of the antibacterial activity of an ethanolic leaf extract of FL at various concentration (50,100 and 200 µg/mL) both gram positive (S.aureus and B.Subtilis) and gram negative (E.coli, P. aeroginosa and P.vulgaris) bacterial species by using disc diffusion method is given in Table (2). It was found that the inhibitive power of the plant against the bacterial species was good at a higher concentration (200µg/mL). On comparing with the standard chloramphenicol , the inhibitive property of the plant Flueggea leucopyrus Willd (FL) has been analyzed. The graphical representation of the inhibition zone length at various concentrations of FL against the bacterial species were depicted in Fig.(2).

Table (2): Antibacterial activity- Zone of inhibition of ethanolic extract of *Flueggea Leucopyrus Willd* (FL)

Bacterial Species	Zone of Inhibition (mm)		
	50 µg/mL	100 µg/mL	200 µg/mL
E.coli	10	12	13
S.aureus	-	9	11
P. aeroginosa	-	-	11
B.subtitis	-	-	9
P. vulgaris	-	-	7

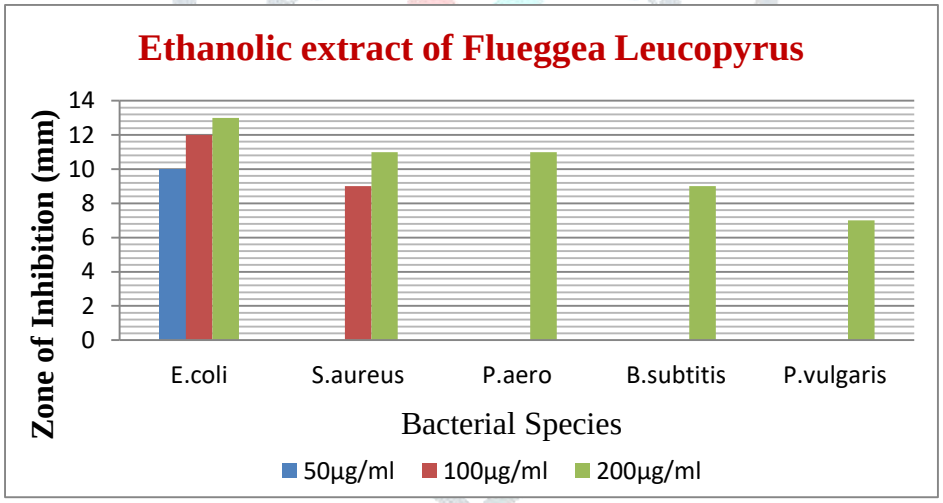


Fig. (2) : Antibacterial activity- Zone of inhibition of ethanolic extract of *Flueggea Leucopyrus Willd* (FL)

3.2.2 Antifungal Activity

The evaluation of antifungal activity of the ethanolic leaves extract of FL at various concentration (50,100 and 200µg/mL) against the fungal species such as A.niger, C.lunata and A.solani by using disc diffusion method. On comparing with the standard Ketoconazole, the antifungal activity of the plant extract of FL shows complete absence of inhibition zone against the fungal species.

3.3 Gas Chromatography- Mass Spectrum Study (GC-MS)

On comparing with the mass spectra from the database of National Institute of Standard Technology (NIST) library, the recorded mass spectra of the GC separated compounds are identified.

The GC-MS chromatogram of the ethanolic leaves extract of *Flueggea leucopyrus Willd* (FL) showed twenty peaks which indicate the presence of twenty chemical constituents (Fig.3). The twenty active constituents with their retention time (RT),

molecular formula, molecular weight (MW) and peak area (%) in the ethanolic leaves extract of *Flueggea leucopyrus Willd* (FL). On comparison of the mass spectra of the constituents with the NIST and WILEY libraries the predominant constituents were characterized and identified. The structure and nature of the compound are presented in Table (3).

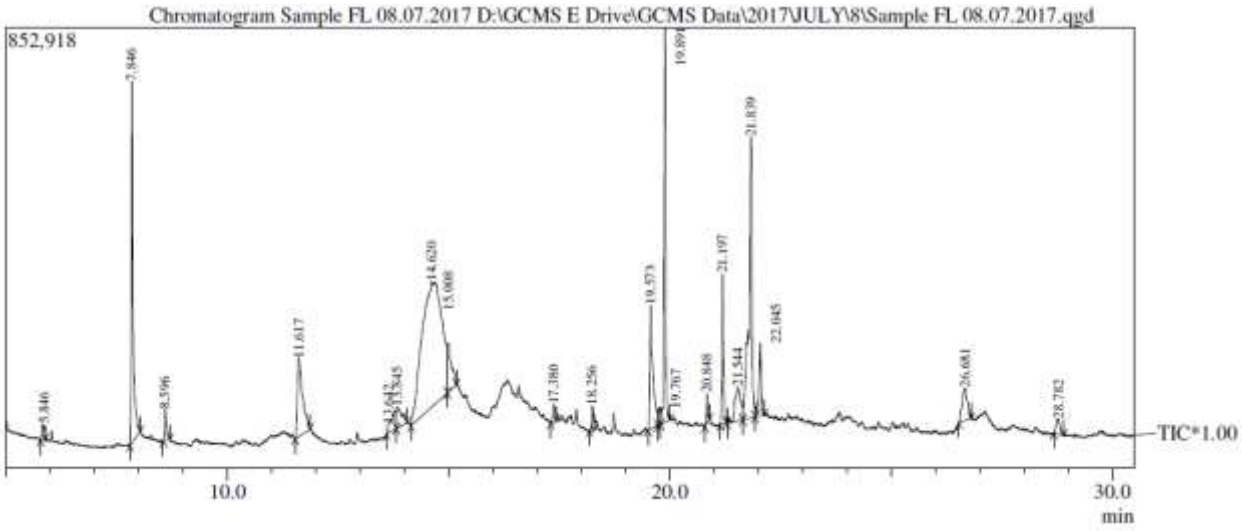


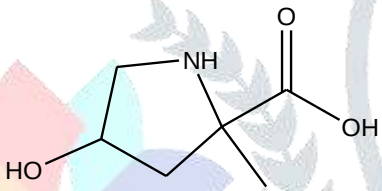
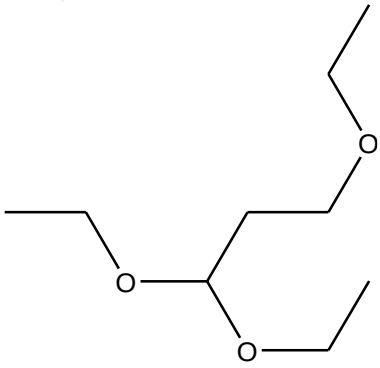
Fig (3): GC-MS chromatogram of the ethanolic leaves extract of *Flueggea Leucopyrus Willd* (FL)

Table (3) – Chemical components identified in the ethanolic extract of the leaves of *Flueggea Leucopyrus Willd* (FL) by GC-MS

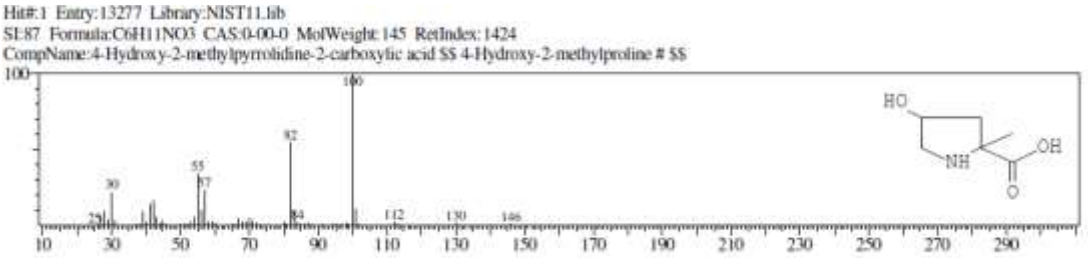
Sl.No.	RT	Name of the Compound	Molecular Formula	Molecular weight	Peak Area %
1	5.846	Pentane, 1,1-diethoxy-	$C_9H_{20}O_2$	160	0.53
2	7.846	Propane, 1,1,3-triethoxy-	$C_9H_{20}O_3$	176	9.37
3	8.596	1-(2-Hydroxyethyl)-1,2,4-triazole	$C_4H_7N_3O$	113	0.96
4	11.617	4-hydroxy-2-methyl-pyrrolidine-2-carboxylic acid	$C_6H_{11}NO_3$	145	5.97
5	13.642	2,2,3,4-tetramethyl- pentane	C_9H_{20}	128	1.53
6	13.845	1-undecene	$C_{11}H_{22}$	154	1.55
7	14.620	4-Hydroxy-2-methylpyrrolidine-2-carboxylic acid	$C_6H_{11}NO_3$	145	36.35
8	15.008	4,5-Heptadien-2-ol, 3,3,6-trimethyl-	$C_{10}H_{18}O$	154	2.72
9	17.380	Tetradecanoic acid	$C_{14}H_{28}O_2$	228	0.64
10	18.256	1-Hexadecyne	$C_{16}H_{30}$	222	0.68
11	19.573	Octadec-9-enoic acid	$C_{18}H_{34}O_2$	282	5.38
12	19.767	Phthalic acid, cis-hex-3-enyl tetradecyl ester	$C_{28}H_{44}O_4$	444	0.54
13	19.891	hexadecanoic acid, ethyl ester	$C_{18}H_{36}O_2$	284	8.60
14	20.848	1-Hexadecanol	$C_{16}H_{34}O$	242	0.66

15	21.197	2-hexadecen-1-ol, 3,7,11,15-tetramethyl-, [R-[R*,R*-(E)]]-	$C_{20}H_{40}O$	296	3.69
16	21.544	(Z,Z)-6,9-cis-3,4-epoxy-nonadecadiene	$C_{19}H_{34}O$	278	2.91
17	21.839	9,12,15-Octadecatrienoic acid, (Z,Z,Z)-	$C_{18}H_{30}O_2$	278	12.22
18	22.045	Octadecanoic acid, ethyl ester	$C_{20}H_{40}O_2$	312	2.36
19	26.681	Squalene	$C_{30}H_{50}$	410	2.49
20	28.782	1,2-benzenedicarboxylic acid, dinonyl ester	$C_{26}H_{42}O_4$	418	0.86

Table-(4): The structure and nature of the predominant chemical components identified in the ethanolic extract of the leaves of *Flueggea Leucopyrus Willd* (FL) by GC-MS

Sl. No.	Name of the Compound	Structure	Nature
1.	4-Hydroxy-2-methylpyrrolidine-2-carboxylic acid		carboxylic acid
2.	Octadecanoic acid, ethyl ester		ester
3.	Propane, 1,1,3-triethoxy-		ethoxide

4.	hexadecanoic acid, ethyl ester		ester



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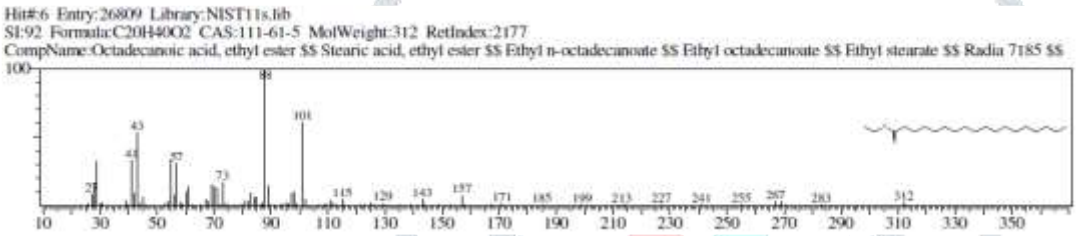

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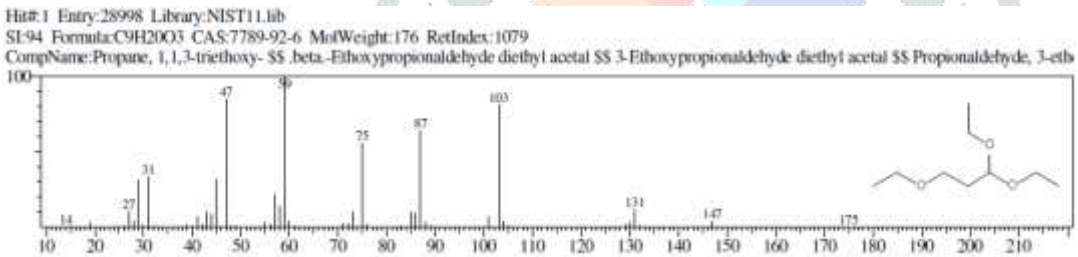

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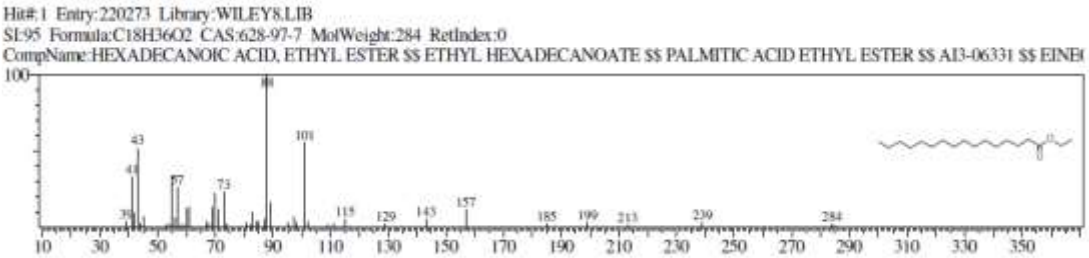


(d)

Fig. (4) (a,b,c,d): Mass Spectrum of predominant chemical constituents present in *Flueggea Leucopyrus Willd* (FL)

3.4 Weight-loss Method

The corrosion rate was determined for carbon steel in 0.5M HCl by using weight loss method. Inhibition efficiency of carbon steel with various concentration of Flueggea leucopyrus Willd (FL) ethanolic leaves extract in 0.5M HCl at room temperature are presented in the Table (5). It is clear from the table that the corrosion inhibition enhances with the inhibitor concentration. The inhibitor systems that are used to determine the corrosion rate and the inhibition efficiency were Zn^{2+}; FL; FL-Zn^{2+}; FL- Zn^{2+}- Additives (Citric acid, tartaric acid and lactic acid). It was found that at FL-Zn^{2+} (50:25) and FL-Zn^{2+} (50:50) the

inhibition efficiencies were 82 and 83% respectively for 72 hours. On further addition of the additives of various concentration (10-50ppm) to the inhibitor ratio of FL-Zn^{2+} (50:50) the inhibition efficiencies of FL-Zn^{2+} - citric acid(40ppm), FL-Zn^{2+} - lactic acid(50ppm), FL-Zn^{2+} - tartaric acid(30ppm), were found to be 86, 87 and 92% respectively.

Table (5): Inhibition efficiencies and corrosion rates of carbon steel in FL-Zn^{2+}, FL-Zn^{2+}-Tartaric acid, FL-Zn^{2+}-Citric acid, FL-Zn^{2+}-Lactic acid, in 0.5M HCl Immersion Period = 72 h

Conc. of FL (ppm)	Conc. of Zn^{2+} ion (ppm)				Conc. of FL-Zn^{2+} ion (ppm)	Conc. of additives (ppm)	Tartaric Acid		Citric acid		Lactic acid	
	25		50									
	IE%	CR (mpy)	IE%	CR (mpy)			IE%	CR (mpy)	IE%	CR (mpy)	IE%	CR (mpy)
10	74	2.1	76	1.9	50:50	10	64	3.6	79	1.7	72	2.1
20	71	2.4	68	2.7		20	73	2.7	72	2.7	38	6.1
30	76	2.0	77	1.9		30	92	0.7	82	1.5	75	2.4
40	79	1.7	80	1.7		40	74	2.1	86	1.3	85	1.4
50	82	1.4	83	1.1		50	90	0.9	75	1.9	87	1.3
Blank		8.5		8.5	Blank			9.9		9.9		9.9

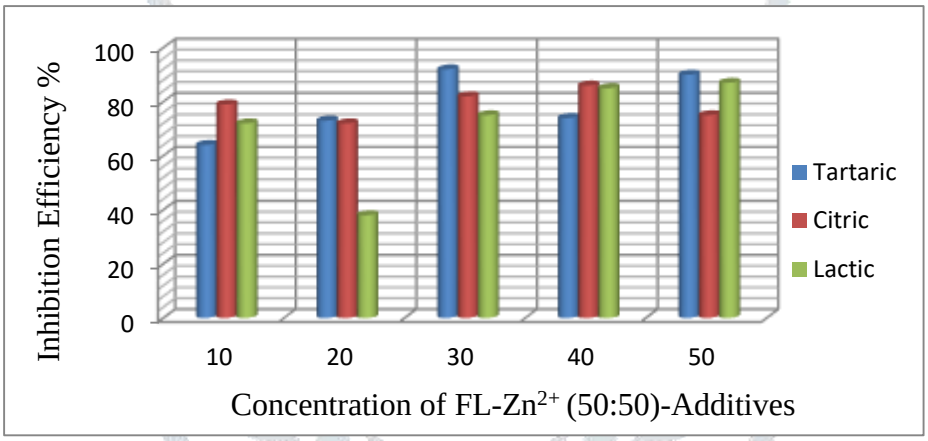


Fig. 5: Inhibition efficiencies of FL-Zn^{2+} (50:50)-Additives (ppm) in various concentration

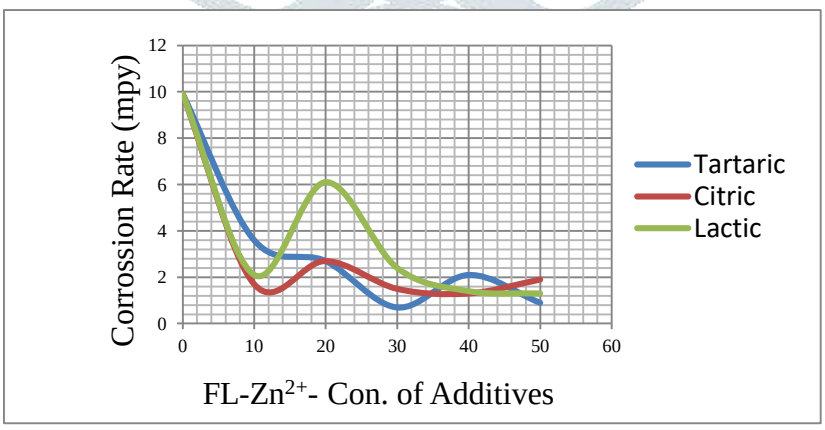


Fig.6: Corrosion Rates of FL-Zn^{2+} (50:50)-Additives (ppm) in various concentration

3.5 Analysis of FTIR

The FTIR spectrum of the extract and the film formed on the surface of the metal immersed in 0.5M HCl in the presence of the inhibitor were taken. FTIR spectroscopy has been used to analyze the protective film formed on the metal surface.[14-15] The FTIR spectrum of the pure extract FL, FL-Zn $^{2+}$, FL-Zn $^{2+}$-Additives(citric, lactic and tartaric acids) as inhibitors are correlated in

Fig.(7). For the pure extract as inhibitor the band observed at 3355.79cm^{-1}. There is a decrease in the frequency from 3600.00 cm^{-1} to 3355.79cm^{-1}. Similar decrease pattern is observed for FL-Zn $^{2+}$, FL-Zn $^{2+}$-Additives(citric, lactic and tartaric acids), the bands were observed at 3364.83, 3359.37, 3384.05and 3441.77 cm $^{-1}$ respectively.

The bands at 1621.64cm-1 and 1278.95cm-1 which are due to the coupling of -C-O stretching and -C-O-H in-plane bending of the carboxylate anion are shifted to 1610.71cm-1 and 1302.30cm-1 in FL-Zn^{2+}. Similar shift in bands were observed in FL-Zn^{2+}-citric acid(1605.19&1278.95cm^{-1}), FL-Zn^{2+}-lactic acid (disappearance of peak & 1415.46cm^{-1}) and FL-Zn^{2+}-tartaric acid(1603.92& 1384.64cm^{-1}). The bands at 1024.69cm-1 and 848.60cm-1 (due to the ring oxygen and metal oxygen bond) are shifted to 1116.88 cm-1 and 851.21cm-1. Similar shift in bands were observed in FL-Zn^{2+}-citric acid(1069.64 & 643.43cm^{-1}), FL-Zn^{2+}-lactic acid (1115.17& 640.32cm^{-1}) and FL-Zn^{2+}-tartaric acid(1051.66& 631.73cm^{-1}). This reveals that due to interaction between the metal and the active constituents there is a change in the chemical nature of the active constituents [16].

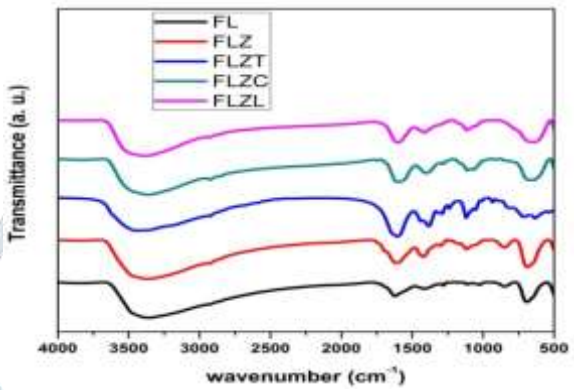
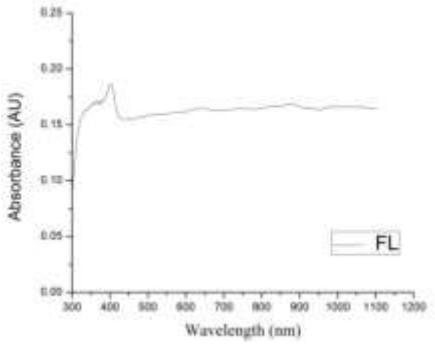


Fig.7: IR Spectra Correlation

3.6 Analysis of UV-Visible absorption spectra

The UV-Visible absorption spectra of the solution containing FL, 50ppm FL – 50ppm Zn^{2+} , (50:50)ppm FL –Zn^{2+}-40ppm Citric acid, (50:50) FL - Zn^{2+} -50ppm lactic acid and (50:50) FL - Zn^{2+} -30ppm tartaric acid are correlated in Fig. 11. A peak appears at 402.50nm (0.1864au), when Zn^{2+} ion is added a peak appears at 373.95nm (0.0555au), the intensity decreases. It is observed that, when additives are added to FL- Zn^{2+} - additives (citric acid, lactic acid, tartaric acid) systems the peak appears at 362.90nm (0.2036au), 372.75 nm (0.1186au), 403.75nm (0.1545au)the intensity decreases on comparing with the FL and FL-Zn^{2+} systems respectively. This indicates the complexation of FL - Zn^{2+} & Additives (Citric, lactic, tartaric acids).



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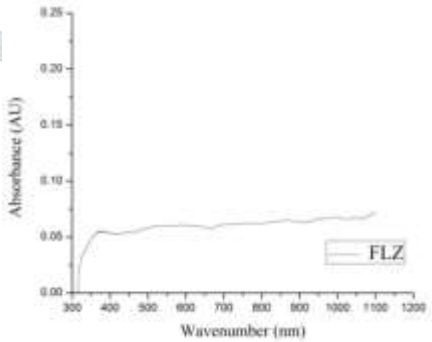

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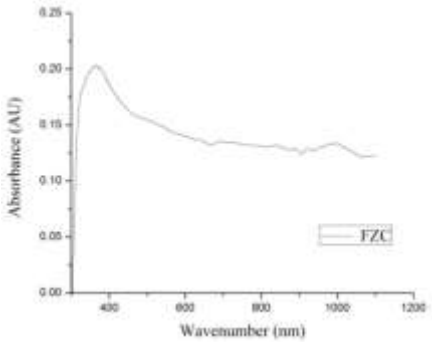

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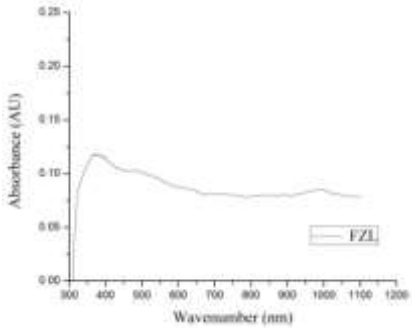

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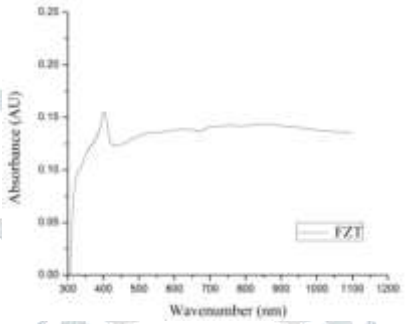


(e)

Fig.8: UV-Visible Spectra

(a) FL (b) FLZ (c) FZC (d) FZL (e) FZT

3.7 Analysis of Fluorescence

Fluorescence spectrum is used to detect the presence of the inhibition complex formed on the metal surface. The λ_{ex} for the emission spectrum of the pure FL as inhibitor is found to be 1006.98, 1006.24 nm and for FL-Zn^{2+} the peak is obtained at 740.36 nm. Fig.9, shows the λ_{ex} for the emission spectrum of the 50ppm FL-50ppm Zn^{2+}-40ppm Citric acid, the peak is obtained at 995.73 nm. The λex for the emission spectrum of the 50ppm FL-50ppm Zn^{2+}-50ppm Lactic acid, the peak is obtained at 541.26 nm. Similarly, for Tartaric acid as additive the peak is obtained at 784.37nm for 30ppm 0f tartaric acid. There is a shift in the intensity on comparing with the pure FL fluorescence value indicates the formation of protective film on the surface of the metal.

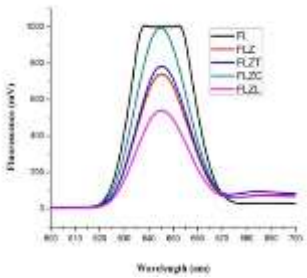
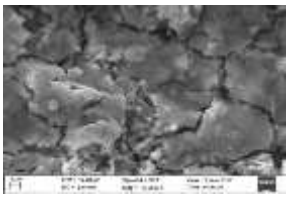


Fig.9: Fluorescence Spectra Correlation

3.8 Scanning Electron Microscope (SEM) Analysis

The texture and pore structure of the inhibited and uninhibited surface in acidic medium are shown in Fig.(10). It is confirmed that the inhibitor systems has formed a dense film over the metal surface.



Blank

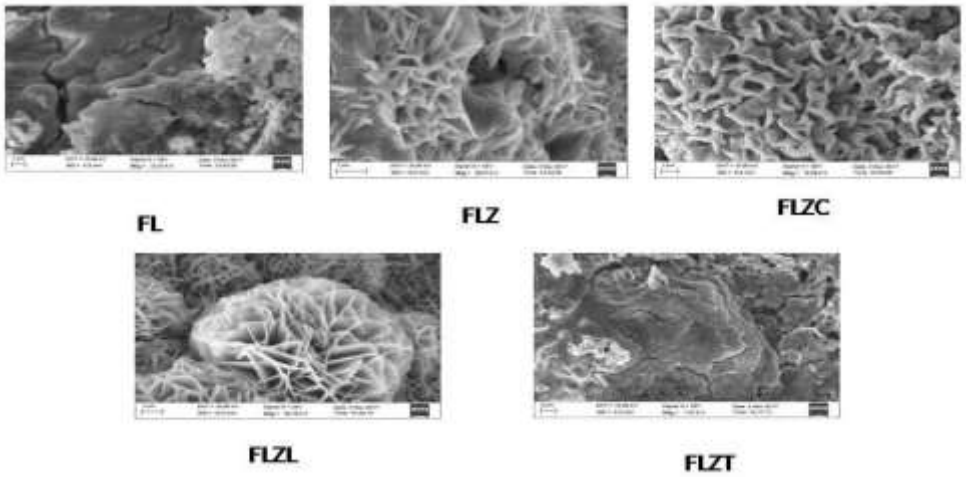
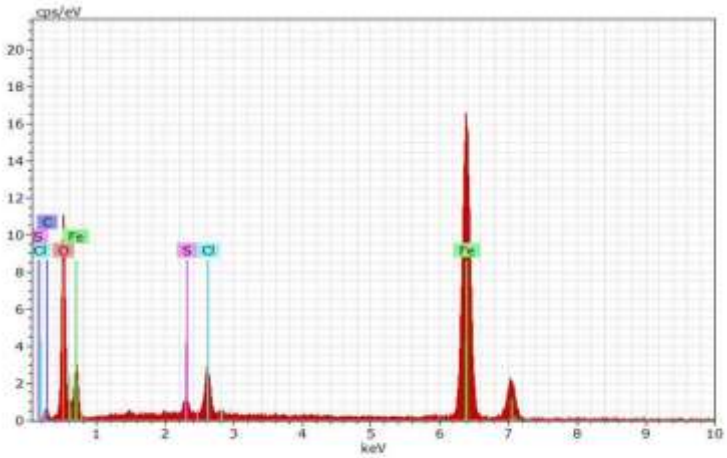


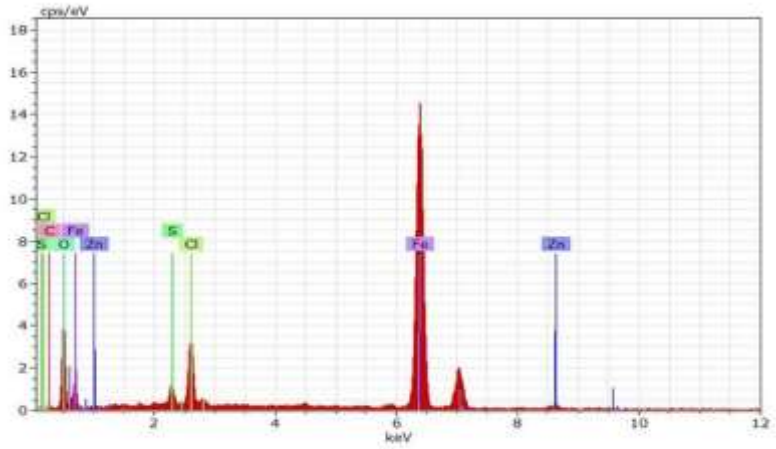
Fig.10: SEM images

3.9 Energy Dispersive Analysis of X-Rays (EDAX)

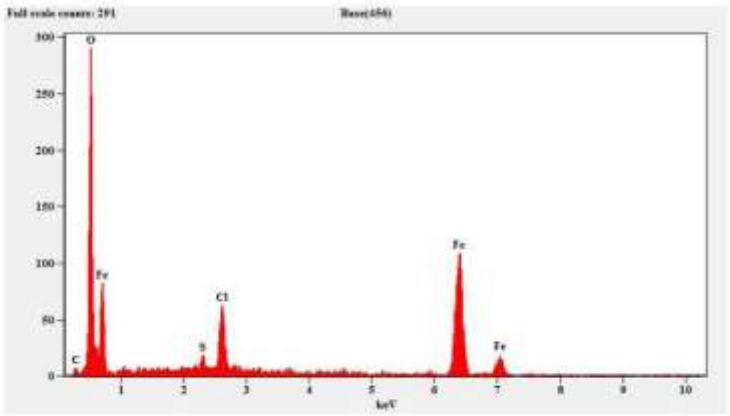
The EDAX spectrum of the resistive film of the inhibitor systems formed on the metal surface shows the characteristic peaks of the elements constituting such as C,O, N, P, S, Fe, and Zn atoms. The EDAX spectrum of carbon steel immersed in well water containing 50 ppm FL and 50 ppm of Zn^{2+} and the additive systems are shown in Fig.(11). It shows the characteristic peak for the existence of OH and Zn. These data show that metal surface is covered the N, C, O and Zn atoms is tabulated in Table-6. Thus the presence of O, N, S and Zn atoms in the resistive film indicates the inhibition efficiency of the inhibitor systems studied.



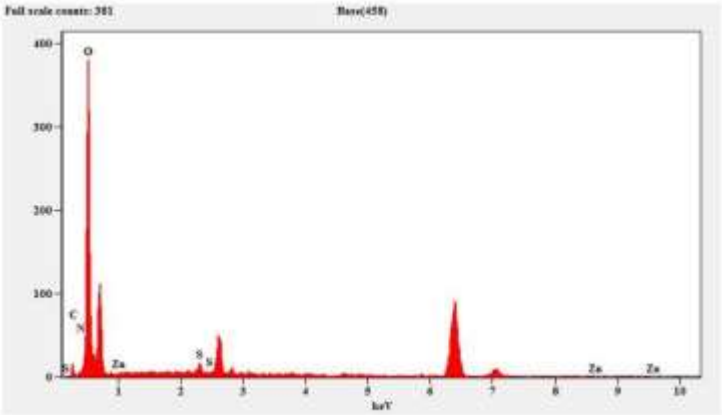
FL


cps/eV
keV
Cl
C
Fe
S
O
Zn
S
Cl
Fe
Zn

FLZ


O
Fe
Cl
C
S
Fe
Fe
keV

FZC


O
C
N
Si
Zn
S
Zn
Zn
keV

FZL

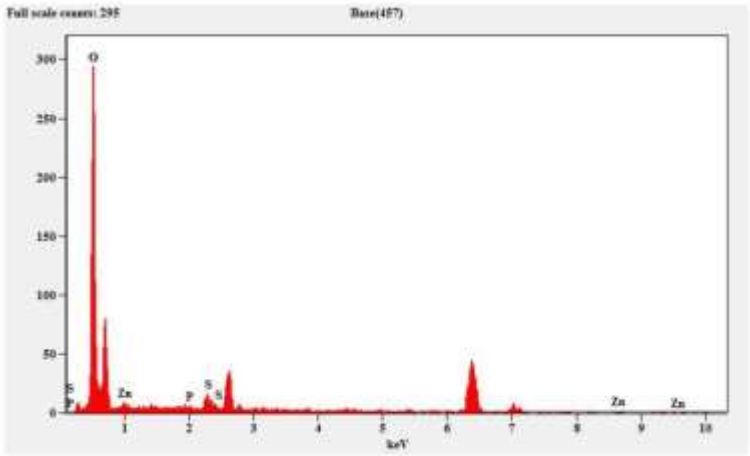


Fig.11: EDAX Spectra

Table-(6): Atomic percentage of the elements present in the resistive film by EDAX

Element	Atomic No.	Atomic %				
		FL	FLZ	FZC	FZL	FZT
C	6	15.69	16.68	16.48	17.38	18.62
N	7	-	-	-	29.34	-
O	8	65.66	52.66	61.32	52.97	77.84
P	15	-	-	-	-	0.19
S	16	0.47	0.09	0.57	0.25	2.78
Cl	17	1.52	3.36	3.35	-	-
Fe	26	16.65	24.89	18.29	-	-
Zn	30	-	0.61	0.25	0.53	0.58

IV. CONCLUSION

From the above study it is concluded that,

- *Flueggea leucopyrus* has a good anticorrosion ability for carbon steel in 0.5 M HCl solution is due to the active phytoconstituents present in the plant.
- The maximum efficiency was found to be 83% at 50ppm FL + 50ppm Zn^{2+}. And the inhibitive efficiency was found to be increased from the maximum efficiency with the additives citric acid (86%), lactic acid (87%) and tartaric acid (92%).
- The shift in the peaks observed in FT-IR, UV-Visible spectra proves the formation of the film on the surface of the metal.
- The variation in the intensities observed in the fluorescence study results the formation of the film on the surface of the metal.
- The protective film formed on the metal surface is found to be denser by the SEM analysis. Thus, the SEM and EDAX images finally confirm the formation of the protective film on the metal surface and the elements present in the resistive film respectively.

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