

Latent Efficacy of Invasive Species (*Parthenium hysterophorus*) for its Anti-Corrosive Propensity

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ABSTRACT: The anti-corrosive property of the Ethanolic extract of *Parthenium hysterophorus* leaves (EEP_hL) was evaluated for Mild Steel (MS) in 0.5 M HCl using chemical method. The experimentation was carried out at room temperature over a variety of inhibitor-concentrations and at various immersion period and the data thus obtained were analyzed to evaluate various corrosion, adsorption and kinetic parameters. The maximum inhibition efficiency was observed 69.45% at 0.13 g/L of EEP_hL. This is attributed to the adsorption of active chemical constituents of *Parthenium hysterophorus* leaves, mainly Parthenin, and Chlorogenic acid, over the MS surface, thus forming a protective film. The adsorptive propensity of the active constituents of EEP_hL was assessed by FT-IR spectroscopy and quantum chemical analysis (QCA). The protective tendency was further elucidated by carrying out surface morphological studies, viz UV-Visible spectroscopy, SEM-EDX and Optical micrography. Thus, EEP_hL is a potential anti-corrosive which can be used to slow down the dissolution of MS in 0.5 M HCl to a significant extent.

KEYWORDS: Corrosion, *Parthenium hysterophorus*, QCA, SEM-EDX, UV-Vis spectroscopy.

I. INTRODUCTION

Many researchers have [1-5] studied the effect of synthetic organic inhibitors on the corrosion of metals in aggressive media. In view of the fact that most of the synthetic inhibitors are hazardous to environment, the use of natural resources as anticorrosive agents are now being explored to be a good replacement of these toxic organic/inorganic inhibitors. Natural resources are rich source of active compounds, such as tannins, amino acids, alkaloids, pigments etc. which has strong adsorptive property. A large number of green corrosion inhibitor for various metals in different medium has been explored [6-10]. The anti-corrosive property of a variety of plant-based inhibitors has also been studied in our laboratory [11-18] and encouraging results were observed. To move a step further, the latent use of those natural resources which themselves are toxic/harmful to the environment, i.e. invasive species, such as *Parthenium hysterophorus* is explored in the present study.

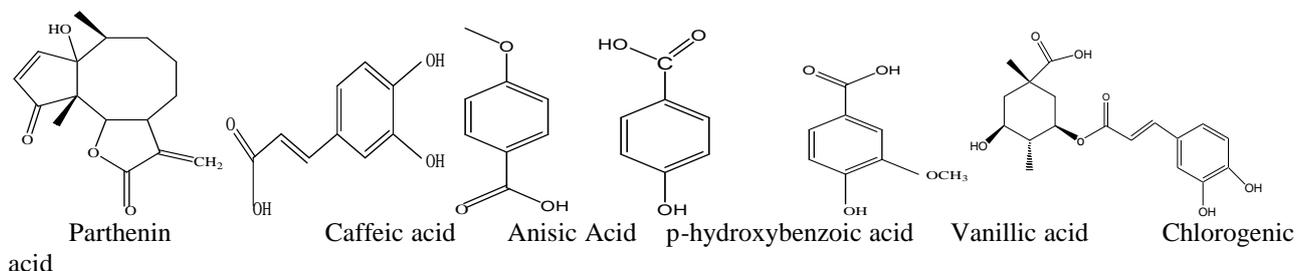
Parthenium hysterophorus, an invasive species, has adverse effects on human health, livestock, agriculture productivity and biodiversity are very well known. Communities living in vicinity of *Parthenium* habitats suffer from allergenic responses, dermatitis, hay fever, and asthma. Livestock productivity (animal health, milk and meat quality, marketing of seeds and grains) is affected either directly by grazing or indirectly by over infestation in grazing pastures. The beneficiary aspects of the extracts of various parts of *Parthenium hysterophorus* plant has been investigated for usages in conventional and traditional system of medicine [19-21].

Literature survey reveal that *Parthenium hysterophorus* consist of **parthenin** (sesquiterpene lactones) in addition to other phenolic acids such as chlorogenic acid, caffeic acid, vanillic acid, ansic acid, panisic acid, parahydroxy benzoic acid etc. These active constituents containing heteroatom such as oxygen and πe^{-s} must have a good tendency of adsorption over metal surfaces [22-27]. Thus, to have 'best out of waste', investigations were carried to explore the anti-corrosive potential of *Parthenium hysterophorus* for the industrially used MS in 0.5 M HCl.

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II. EXPERIMENTATION

Chemical measurements

The Chemical measurements were carried out by the standard procedure as reported earlier [11-18]. MS coupons were totally immersed in solutions having 0.5 M HCl and various concentrations of the studied inhibitor (*Parthenium h.*). The inhibitive tendency was determined by weight loss of the coupons for various time intervals at room temperature using an analytical balance (Adair Dutt 205 ACS). The experimentation was carried out in triplet and average weight loss was recorded.

Specimen preparation:

Rectangular specimen (coupons) of MS having the composition: (Mn) 0.3%, 0.01% (Mo), 0.039% (Cr), Fe 99.7%, as analyzed by XRF studies (Fig. 1), were prepared by mechanically press-cut each of dimensions (3 cm x 2.4 cm x 0.16 cm) with a hole about 0.12 mm diameter drilled at one end for free suspension. Each coupon was surface-treated, degreased with acetone & washed with distilled water and preserved in desiccators, as per standard procedures [11-18].

Preparation of ethanolic extract of leaves of the invasive species:

Ethanolic extract of *Parthenium hysterophorus* (EEPh) was obtained by refluxing the dried plant parts in Soxhlet extractor as per standard procedures reported earlier [11-18]. The plant material extracted was found to be 6.5 g/L.

Preparation of test solutions:

0.5 M HCl was prepared using AnalR grade of HCl with triply distilled water. Test solutions were prepared by adding EEPh to 0.5 M HCl in increasing concentration range 0.0065 to 0.013 g/L in 0.5 M HCl.

III. CHARACTERIZATION & SURFACE MORPHOLOGICAL ANALYSES

1. Quantum Chemical Analysis:

QCA were carried out by using the PM3 method of the Quantum Chemical Package MOPAC 6.0 of Hyperchem 7.0. The energy of the highest occupied molecular orbital (E_{HOMO}), the energy of the Lowest unoccupied molecular orbital (E_{LUMO}), energy band gap, $\Delta E = E_{\text{HOMO}} - E_{\text{LUMO}}$, heat of formation and the dipole moment (μ) were evaluated.

2. UV-Visible Spectroscopic Analysis:

The adsorption behaviour of active constituents of the extract was analysed by UV-Visible spectras (8400 Shimadzu, Japan, quartz glass cell, 400-800 nm visible range).

3. Surface Morphological Analyses:

SEM-EDX and Optical Micrographs:

Surface morphology of MS coupon-surface was studied by carrying out SEM-EDX (ZEISS - in the range of 10 μ) and Optical Micrographs (LABOMED microscope in the range of 20 μ).

IV. RESULTS AND DISCUSSION

The purity of the coupon is clearly indicated by XRF graph (Fig. 1). From the weight loss data various corrosion parameters, like corrosion rate (ρ_{corr}) (mm^{-1}), percentage inhibition efficiency (IE %), fractional surface coverage (θ), adsorption equilibrium constant (K_{ad}) etc. were evaluated and tabulated in Table 1. Fig. 2 shows the linear variation of corrosion rate with increasing concentrations of EEPh at various immersion periods for the acid corrosion of MS in 0.5

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M HCl at room temperature (303±1K). The rate of corrosion was significantly suppressed in presence of inhibitor (EEPh) as compared with free acid.

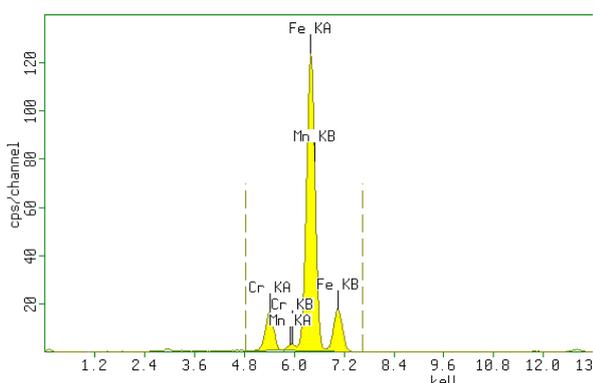


Fig. 1: XRF graph of polished MS Coupon and with EEPh for MS

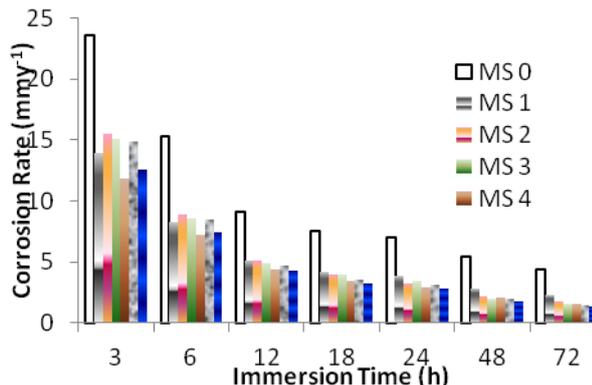


Fig. 2: Corrosion rate v/s immersion time without

Increase in I.E. % with increasing concentration of EEPh shows the inhibitive propensity of the extract (Fig. 3). The maximum IE% was found to be 69.45% at maximum concentration of EEPh (0.13 g/L) at 72 h.

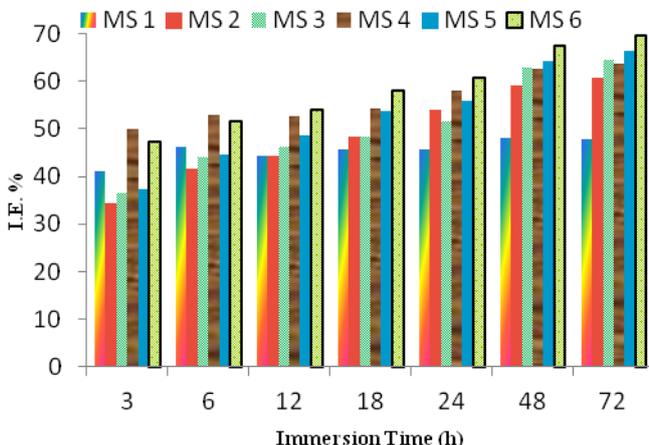


Fig. 3 Inhibition efficiency (%) of EEPh v/s immersion time (h) concentration (0.13 g/L)

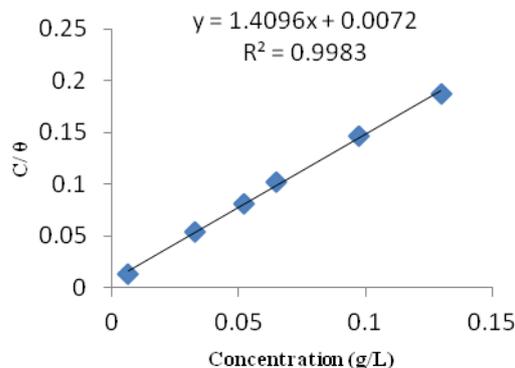


Fig.4: C/θ v/s EEPh

The corrosion inhibition may be attributed to adsorption of active constituents of *Parthenium hysterophorus* as they contain heteroatoms like oxygen which get protonated in the acidic medium and positively charged inhibitor molecule adsorbed on the negatively charged metal surface via electrostatic force of attraction and also due to π - electrons interaction with metal.

Adsorption Isotherms

The dependency of the surface coverage (θ) was found as a function of the concentration of the additive (C). A direct relationship is assumed between EEPh concentration and the surface coverage of the inhibitor may be expressed as following:

$$C/\theta = 1/K + C \quad \dots\dots\dots (1)$$

K_{ad} is the adsorption equilibrium constant which was calculated from the intercepts of the straight line C_{inh}/θ -axis. The plot of C/θ vs. concentration at room temperature results in straight line with high linear regression correlation coefficient (Fig.4). The slope was also found almost unity thus following Langmuir adsorption isotherm which indicate

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a strong adherence of the adsorption process to the formation of the monolayer of the inhibitor molecules on to MS surface [22-26]. The interaction between adsorbed molecules and metal surface was further described by the free energy of adsorption ΔG°_{ads} calculated by the help of expression: $\Delta G^{\circ} (kJ) = - RT \ln (K_{ad} \times 55.5)$ (2) where, K_{ad} = adsorption equilibrium constant; T = absolute temperature (K); R = $8.314 \text{ JK}^{-1} \text{ mol}^{-1}$

Table 1: Corrosion parameters of acid Corrosion of MS without & with various concentrations of EEP*h* at various immersion time (h) at room temperature $303 \pm 1 \text{ K}$

Time (h)	EEPh Concentration (g/L)	Corrosion parameters					
		Weight loss (mg)	Corrosion rate (ρ_{Corr}) $\times 10^{-1}$	Inhibition efficiency (IE %)	Functional surface coverage (Θ) $\times 10^2$	Adsorption Equilibrium Constant (K_{ad})	Gibbs free energy (ΔG) KJ mol^{-1}
3	MS-0 0.0000	102	23.68	--	--	--	--
	MS-1 0.0065	60	13.93	41.18	41.18	107.69	-21.91
	MS-2 0.0325	67	15.55	34.31	34.31	16.07	-17.11
	MS-3 0.052	65	15.09	36.27	36.27	10.95	-16.15
	MS-4 0.065	51	11.84	50.00	50.00	15.38	-17.00
	MS-5 0.0975	64	14.85	37.25	37.25	6.09	-14.67
	MS-6 0.13	54	12.53	47.06	47.06	6.84	-14.96
6	MS-0 0.0000	132	15.32	--	--	--	--
	MS-1 0.0065	71	8.24	46.21	46.21	132.18	-22.42
	MS-2 0.0325	77	8.94	41.67	41.67	21.98	-17.90
	MS-3 0.052	74	8.59	43.94	43.94	15.07	-16.95
	MS-4 0.065	62	7.19	53.03	53.03	17.37	-17.31
	MS-5 0.0975	73	8.47	44.70	44.70	8.29	-15.45
	MS-6 0.13	64	7.43	51.51	51.51	8.17	-15.41
12	MS-0 0.0000	158	9.17	--	--	--	--
	MS-1 0.0065	88	5.11	44.30	44.30	122.38	-22.23
	MS-2 0.0325	88	5.11	44.30	44.30	24.48	-18.17
	MS-3 0.052	85	4.93	46.20	46.20	16.52	-17.18
	MS-4 0.065	75	4.35	52.53	52.53	17.03	-17.26
	MS-5 0.0975	81	4.70	48.73	48.73	9.75	-15.85
	MS-6 0.13	73	4.24	53.80	53.80	8.96	-15.64
18	MS-0 0.0000	197	7.62	--	--	--	--
	MS-1 0.0065	107	4.14	45.69	45.69	129.40	-22.37
	MS-2 0.0325	102	3.94	48.22	48.22	28.66	-18.57
	MS-3 0.052	102	3.94	48.22	48.22	17.91	-17.39
	MS-4 0.065	90	3.48	54.31	54.31	18.29	-17.44
	MS-5 0.0975	91	3.52	53.81	53.81	11.95	-16.37
	MS-6 0.13	83	3.21	57.87	57.87	10.56	-16.06
24	MS-0 0.0000	243	7.05	--	--	--	--
	MS-1 0.0065	132	3.83	45.68	45.68	129.37	-22.37
	MS-2 0.0325	112	3.25	53.91	53.91	35.99	-19.15
	MS-3 0.052	118	3.42	51.44	51.44	20.37	-17.71
	MS-4 0.065	102	2.96	58.02	58.02	21.27	-17.82
	MS-5 0.0975	107	3.10	55.97	55.97	13.04	-16.59
	MS-6 0.13	96	2.78	60.49	60.49	11.78	-16.33
48	MS-0 0.0000	374	5.42	--	--	--	--
	MS-1 0.0065	194	2.81	48.13	48.13	142.74	-22.62
	MS-2 0.0325	153	2.22	59.09	59.09	44.44	-19.68
	MS-3 0.052	139	2.02	62.83	62.83	32.51	-18.89

	MS-4 0.065	140	2.03	62.57	62.57	25.71	-18.30
	MS-5 0.0975	134	1.94	64.17	64.17	18.37	-17.45
	MS-6 0.13	122	1.77	67.38	67.38	15.89	-17.09
72	MS-0 0.0000	455	4.40	--	--	--	--
	MS-1 0.0065	238	2.30	47.69	47.69	140.27	-22.57
	MS-2 0.0325	179	1.73	60.66	60.66	47.44	-19.84
	MS-3 0.052	162	1.57	64.39	64.39	34.78	-19.06
	MS-4 0.065	165	1.59	63.73	63.73	27.04	-18.42
	MS-5 0.0975	153	1.48	66.37	66.37	20.24	-17.70
	MS-6 0.13	139	1.34	69.45	69.45	17.49	-17.33

The negative values of ΔG°_{ads} (Table 1) clearly indicate the spontaneity of adsorption of the inhibitor molecules as well as strong interface between adsorbed molecules and metal surface. Usually, values of ΔG°_{ads} up to -20 kJ mol^{-1} are consistent with electrostatic interaction between charged molecules and a charged metal (which indicates physical adsorption) while those more negative than -40 kJ mol^{-1} involves charge sharing or transfer from the inhibitor components to the metal surface to form a coordinate type of bond (which indicates chemisorptions). The reported values for the discussed inhibitor clearly states that physical adsorption takes place [28].

Quantum Chemical Analysis:

The Quantum Chemical parameters of the active constituent of *Parthenium hysterophorus* (Parthenin) were evaluated for the optimized structure, 3D structure of total charge density (Fig. 5 a – d) and were tabulated (Table 2).

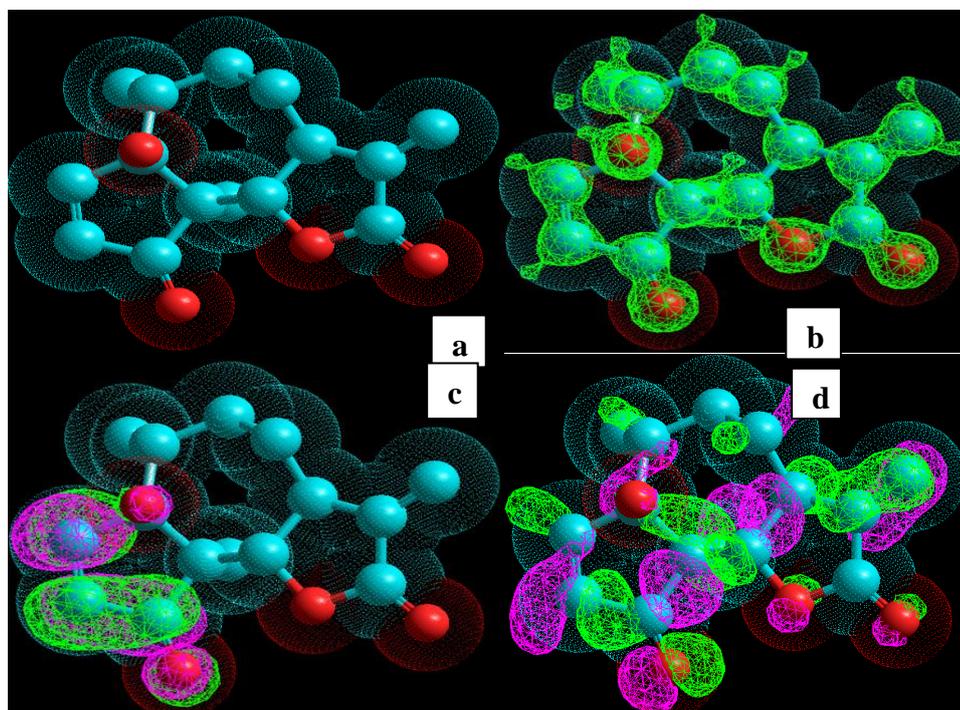


Fig. 5 (a-d): (a) The optimized structure of Parthenin, (b) E_{HOMO} , (c) E_{LUMO} , (d) 3D structure of total charge density

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Tab 2: Quantum Chemical Parameters of active constituent of *EEPh* (Parthenin)

Plant constituents	Total Energy (kcal/mol)	Dipole Moment (Debye)	E_{HOMO} (eV)	E_{LUMO} (eV)	$\Delta E = E_{HOMO} - E_{LUMO}$
Parthenin	-108.461	8.319	-10.291	-0.223	-10.068

For the prediction of active sites and the reactivity of the chemical species E_{HOMO} and E_{LUMO} are the remarkable parameter as they deal with the electron donating and accepting tendency of the molecule. The lower values of energy band gap ($\Delta E = E_{HOMO} - E_{LUMO}$) implies that the energy required to remove an electron from last occupied molecular orbital is minimum that is responsible for the enhancement of inhibition efficiency. A potential inhibitor has tendency to donate or accept electrons or bind on metal surface. The results seem to indicate that both the values of the energy band gap, ΔE as well as that of the dipole moment, μ favours the potential of parthenin as corrosion inhibitor. The negative value of the heat of formation of the *EEPh* states that the inhibitor forms a very stable complex on metal surface and its formation is spontaneous [28].

UV-visible Spectroscopic Analysis:

Adsorption of *EEPh* on MS forms a film of metal-inhibitor complex that can be illustrated by carrying UV-Visible Spectroscopy. UV-Visible absorption spectras for extract and that of MS immersed in 0.5 M HCl with 0.13 g/L *EEPh* (Fig. 6) indicate the absorbance band at 600-700 nm due to the presence of highly conjugated aromatic system in the plant constituents and are responsible for $\pi-\pi^*$ transition. Some researchers also suggested that these peaks are consistent with d-d transition [29-30]. In the presence of MS in test solution 3 new bands appear in the region of 650 to 550 nm thus indicating the formation of a *EEPh*-MS complex between two species in solution.

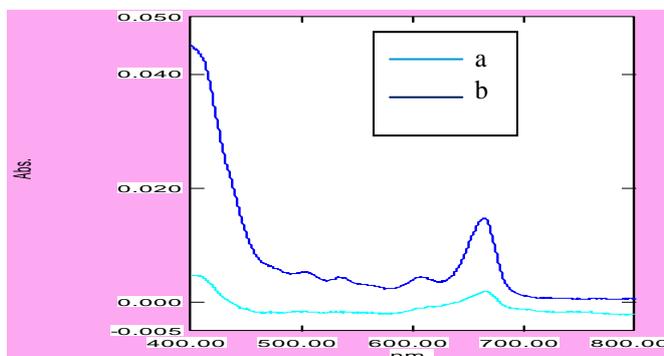


Fig. 6: UV-visible spectras of (a) the *EEPh* and (b) the test solution with MS immersed in *EEPh* (0.13 g/L) for 72 h.

Surface Morphological Analyses:

Optical Micrographs :

Fig 7 (a and b) shows the Optical Micrographs of MS in aggressive media without and with inhibitor. Fig 7(a) reveal the rough corroded metal surface with pit in aggressive media without inhibitor while Fig. 7(b) reveal the smooth surface with reduced number of cracks and pits in presence of inhibitor [18].

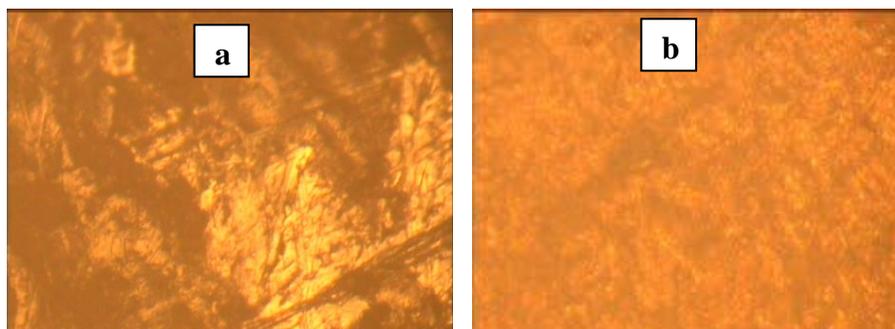
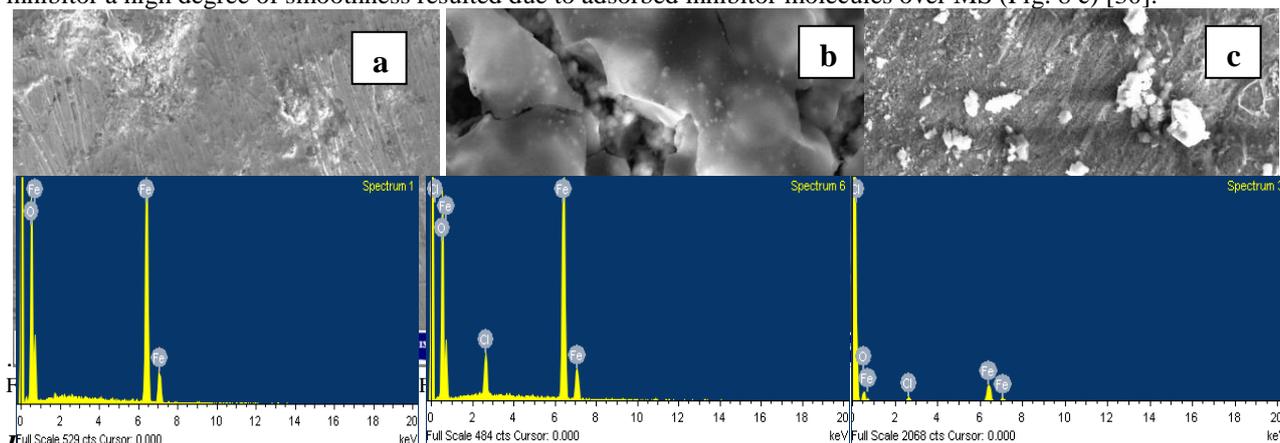


Fig. 7(a-b): Optical micrograph of the MS in aggressive media without EEPh with EEPh.

SEM-EDX Analysis:

Figs. 8 (a) illustrated the surface treated MS surface with scratches on it. Fig 8 (b) illustrated flakes owing to corrosion products where the surface is strongly damaged by corrosion in aggressive media without inhibitor. In the presence of inhibitor a high degree of smoothness resulted due to adsorbed inhibitor molecules over MS (Fig. 8 c) [30].



Energy dispersive X-ray (EDX) analysis of MS coupons was also carried out mainly to estimate the percentage of chloride content present on the surface of metal coupon in the absence and presence of optimum concentration of inhibitor. Fig. 9 (a) revealed that the surface treated MS coupon was chlorine free. A significant amount of chloride ions were found on the coupons immersed in HCl solution (Fig. 9 b) which may be due to the aggressive attack of the chloride ions. Fig. 9 (c) depicted the spectrum of MS immersed with EEPh (0.13 g/L) in 0.5 M HCl for 72 h. Almost negligible amount of chloride ions were found on the surface of MS compared to the MS treated with 0.5 M HCl as well as the peaks for Fe get reduced due to adsorption of inhibitor molecules on MS surface [30].



Fig. 10: EDX spectrum of MS coupons in 0.5 M HCl (a) surface treated; (b) without inhibitor; and (c) with 0.13 g/L EEPh.

V. CONCLUSIONS

Parthenium hysterophorus was found to mitigate acid corrosion of MS quite significantly. The maximum inhibition efficiency (69.45%) was attained after 72 hours of immersion in 0.13 g/L of EEPh at room temperature. The inhibitive potency is by virtue of adsorption of phytochemicals present in *Parthenium hysterophorus* extract over metal surface an

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it was found to best fitted in Langmuir adsorption isotherm. The free energy of adsorption specifies the strong and spontaneous adsorption of the extracts on the MS surface. Quantum Chemical Analysis elucidated the adsorptive mode of molecules present in *Parthenium hysterophorus*. The spectroscopic studies (UV-Visible spectroscopy) found to complement the experimental results and endorse the adsorption of inhibitor over metal surface. The SEM-EDX and Optical Micrographs also reveal the adsorption of inhibitor molecules over metal thus protecting from corrosion in acid medium.

From the above data and studies it can be concluded that a invasive plant *Parthenium hysterophorus* can be used as good and effective corrosion inhibitor.

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