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ANTI-CORROSION ACTIVITY OF *EUGENIA SINGAMPATTIANA* BEDDOME *LEAVES* ON API – 5L – GRADE – X60 STEEL IN CRUDE OIL WITH 10%, FREE WATER ENVIRONMENT

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ABSTRACT

Extract of *Eugenia Singampattiana Beddome* has shown an excellent inhibition performance on API – 5L – Grade – X60 Steel in Crude oil with 10%, Free water environment. The inhibition efficiency increased with the increase of inhibitor concentration and attain of maximum 99.17% inhibition efficiency. The multilayer adsorption on the metal surface may follows physisorption mechanism. Value of activation energy (E_a), enthalpy of adsorption (ΔH_{ads}) and free energy changes (ΔG_{ads}) clearly suggests that the adsorption of inhibitor on metal surface follows Physisorption, endothermic and spontaneous process respectively. The inhibitor is found to obey Langmuir adsorption isotherms.

KEYWORDS: API – 5L – Grade – X60 Steel, Green inhibitor, Acid, Mass Loss, Adsorption.

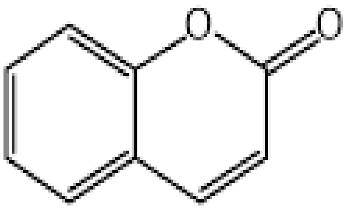
1.0. INTRODUCTION

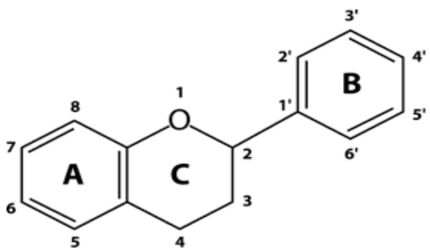
Mild steel is most familiar material widely employed almost all the field in world wide. But the facing main problem of using this material undergoes dissolution in acidic environments. In industry, for cleaning processes, mostly acid solutions are commonly used for the removal of rust and scale. Use of inhibitor is one of the best method to prevent metal dissolution is very common.[1,3] Most of the well-known acid inhibitors are organic compounds containing hetero atoms viz; nitrogen, sulfur, oxygen, heterocyclic compounds with a polar functional group and conjugated double bond.[4,5] These kinds of compounds are adsorbed on the metallic surface and block the active corrosion sites. Most of the synthetic chemicals are costly, toxic to both human being and the environment. To overcomes, these difficulties nowadays, most of the researchers, choosing the inhibition are plenty, cheap, nontoxic and environmentally friendly natural products as corrosion inhibitors. These natural organic compounds are either separated or extracted from aromatic herbs, spices and medicinal plants. Plant extracts are an incredibly rich source of naturally synthesized chemical compounds that can be extracted by simple procedures with low cost and are biodegradable in nature. The plant extract are rich sources of molecules which have appreciably high inhibition efficiency and hence termed as "Green Inhibitors". These inhibitors do not contain heavy metals or other toxic compounds. Recent studies using plants containing heteroatom such as oxygen, nitrogen and sulphur *like Ocimum viridis, Phyllanthus amarus, Annona squamosal, Argan, Psidium guajava, black pepper, Punica granatum, Mentha pulegium, Cnidoscolus chayamans, Solanum Torvum, Pisonia Grandis, mimusops elengi, Sauropus Androgynus, Kingiodendron pinnatum, Wrightia Tinctoria, Aloe- Vera gel, Hibiscus Rosa Sinensis and Azadirachita Indica leaves, Aihagi Maurerum and Merusnigra and Apricot Leaves, Adansonia digitata (Baobab) fruit pulp and seeds, Cupressus sempervirens, Feungreak leaves, essential oils of Alpinia Galanga, Chrysophyllum Albidum leaves, poupartiabirrea back, Acacia Tortilis, Arabinogalactan, keto sulphone drug, Artemisia Mesatlantica essential oil, spirogyraalgae, Tragacanth gum, Prunus Persic, Lemon Gross, Secang heartwood extract (Caesalpinia sappan I), Dried marjoram leaves*[6,39] have also been used for inhibition of corrosion. In continuous of our research work, the present investigation is the *Eugenia Singampattiana Beddome* leaves extract used as corrosion inhibitor on API – 5L – Grade – X60 Steel in 1.0N Hydrochloric acid have been investigated with various periods of contact and temperature using the mass loss measurements.

2.0 MATERIALS AND METHODS

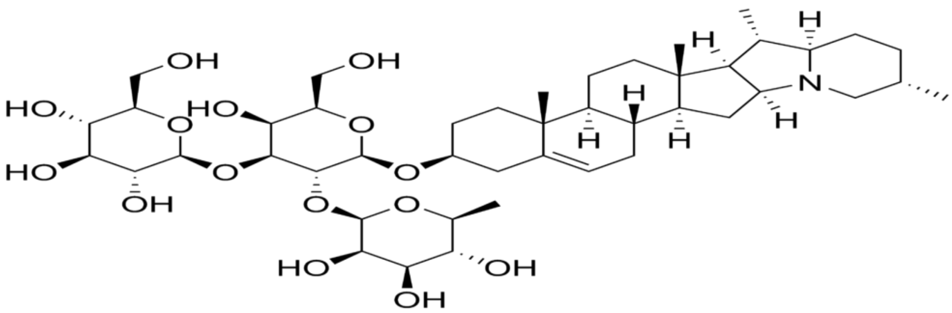
2.1 Properties of *Eugenia Singampattiana Beddome leaves*

Eugenia singampattiana Bedd. is a critically endangered medicinal tree, endemic to the tail end of Southern Western Ghats, and this species is highly restricted to evergreen patches of Agasthyamalai hills. It is proven as anticancerous, antitumerous, antioxidative, antimicrobial, antifungal, antiinflammatory, antihyperlipidaemic and antidiabetic agents. The tribal people have enormous indigenous knowledge on this particular species which is used for food and medicinal purposes effectively. *Ex-situ* and *in-situ* conservation strategies are to be developed for this particular species by protecting the existing natural strands and through species specific multiplication and restoration programme. Compounds like flavanol glycosides, polyphenols, ellagic acids, gallic acids were reported earlier from various species of Eugenia and GC-MS analysis of leaves have proved the presence of eighteen compounds. The major identified compound are 5-Methoxy-2,2,6-trimethyl-1(3-methylbuta-1,3-dienyl)-7-oxa-bicyclo heptanes followed by 1,2,3-Benzenetriol (pyrogallol), α-caryophyllene, 2-propen-1-one, 1-(2,6-dihydroxy-4-ethoxyphenyl)3-phenyl, n-Hexadecanoic acid, 9,12-Octadeca dienoic acid, 2-pentanone, 1-(2,4,6-trihydroxyphenyl)α- Amyrin (β-amyrin), Squalene and limonene**.** The other compounds like alkaloids, coumarins, catechins, glycosides, flavanoids, phenols, steroids, saponins, tannins, terpenes, sugars, xanthoproteins, derivatives and fixed oils are also reported from *E. singampattiana.* Several studies have proved the significant anti-hyper proteinemia, anti-diabetic, anti-oxidant, anti-inflammatory and anti-hyper lipidaemic effects of this species. Flavonoids are also reported to regenerate the damaged pancreatic beta cells and phenols have found tube effective anti-hyperglycemic agents.





Coumarin Flavanoids



Saponins

Figure 1: Chemical structure of the main active compounds present in *Eugenia Singampattiana Beddome* leaves extract.

Figure 2: Plant of *Eugenia Singampattiana Beddome*.

2.2 Stock solution of *Cansjera Rheedii Leaves* Extract

*Eugenia Singampattiana Beddome l*eaves (ESBL) was collected from the source and dried under shadow for about 10-15 days, grained well, then soaked in a solution of ethyl alcohol for about 48 hrs, Then it is filtered followed by evaporation in order to remove the alcohol solvent completely and the pure plant extract was collected. From this extract, different concentration of 10 to 1000ppm stock solution was prepared using double distilled water and used throughout our present investigation.

2.3 Specimen preparation

Rectangular specimen of API – 5L – Grade – X60 Steel was mechanically pressed cut to form different coupons, each of dimension exactly 20cm^2 (5x2x2cm) with emery wheel of 80 and 120 and degreased with trichloroethylene, washed with distilled water, cleaned and dried, then stored in desicators for our present study.

2.4 Mass loss method

In the mass loss measurements on API – 5L – Grade – X60 Steel in triplicate were completely immersed in 100ml of the test solution in the presence and absence of the inhibitor. The metal specimens were withdrawn from the test solutions after 24 to 360 hrs exposure at room temperature and also measured temperature ranges from 313K to 333K. The Mass loss was taken as the difference in weight of the specimens before and after immersion using digital balance with sensitivity of ±1 mg. The tests were performed in triplicate to guarantee the reliability of the results and the mean value of the mass loss is reported. From the mass loss measurements, the corrosion rate was calculated using the following relationship.

$$\text{Corrosion Rate}(\text{mmpy}) = \frac{87.6\times W}{DAT} \quad \text{---------------------} \ (1)$$

[Where, mmpy = millimeter per year, W = Mass loss (mg), D = Density (gm/cm^3), A = Area of specimen (cm^2), T = time in hours]

The inhibition efficiency (%IE) and degree of surface coverage (θ) were calculated using the following equations.

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

$$\theta = \frac{W_1 - W_2}{W_1} \quad \text{-------------------------------} \ (3)$$

(Where W_1 and W_2 are the corrosion rates in the absence and presence of the inhibitor respectively).

3.0 RESULTS AND DISCUSSION

3.1 Effect of time variation

Anti-Corrosion behavior of API – 5L – Grade – X60 Steel in 1.0N HCl containing the presence and absence of ESBL extract with various exposure times (24hrs to 360 hrs) are shown in Table-1**.** Observed values are clearly indicates that the in presence of ESBL extract, the corrosion rate moderately decreased from 1.4036 to 0.0117 mmpy for 24 hrs and 0.0532 to 0.0055 mmpy after 360 hrs with increase of inhibitor concentration (0 to 1000 ppm). The maximum of 99.17% of inhibition efficiency is achieved after 24 hrs exposure time, suggests that the adsorption process occurs mainly due to the presence of active phytochemical constituents present in the inhibitor molecule especially hetero atom containing species and the metal ion from the surface of the metal. However beyond this exposure time, the adsorbed layer may slightly destabilized upto 120hrs, the %IE deviated from 99% to 80%, but after that the inhibition efficiently increased to 89.71% even then 360hrs time means that the inhibitor is more effective only 1000ppm concentration.

Table 1: The corrosion parameters of API – 5L – Grade – X60 Steel in 1.0N HCl containing different concentration(0 to 1000ppm) of ESBL extract after 24 to 360 hours exposure time.

Conc. of inhibitors (ppm)	24 hrs		72 hrs		120 hrs		240 hrs		360 hrs	
	C.R (mmpy)	% I.E	C.R (mmpy)	% I.E	C.R (mmpy)	% I.E	C.R (mmpy)	% I.E	C.R (mmpy)	% I.E
0	1.4036	-	0.1248	-	0.1096	-	0.0561	-	0.0532	-
10	0.0818	94.17	0.0350	71.88	0.0351	68.09	0.0374	33.33	0.0117	77.94
50	0.0711	95.00	0.0350	71.88	0.0327	70.21	0.0211	62.50	0.0109	79.91
100	0.0351	97.50	0.0320	75.00	0.0304	72.34	0.0164	70.83	0.0100	80.88
500	0.0332	98.33	0.0234	81.25	0.0280	74.47	0.0152	72.92	0.0084	83.82
1000	0.0117	99.17	0.0156	87.50	0.0211	80.85	0.0094	83.33	0.0055	89.71

3.2. Effect of Temperature

Dissolution behavior of API – 5L – Grade – X60 Steel in 1.0N HCl containing various concentration of ESBL extract at various temperature 303to 333K and the observed values are listed in Table-2. The observed results reveals that the corrosion rate decreased with increase of inhibitor concentrations and decreased with rise in Temperature from 303 to 333K. The maximum of

97.22% inhibition efficiency is achieved at 303K. However the value of inhibition efficiency is decreased with rise in temperature may suggests that the process is Physisorption mechanism due to multilayer film formation on the metal surface.

Table 2: The corrosion parameters of API – 5L – Grade – X60 Steel in 1.0N HCl containing different concentration of ESBL extract at 313 to 333 K after one hour exposure time.

Conc. of inhibitor (ppm)	303 K		313 K		333 K	
	C.R (mmpy)	% I.E	C.R (mmpy)	% I.E	C.R (mmpy)	% I.E
0	10.1060	-	14.5996	-	11.6675	-
10	1.1229	88.89	11.5096	21.15	5.9831	15.79
50	0.8422	91.67	8.9831	38.46	6.7373	36.84
100	0.5614	94.44	5.8752	59.62	6.1758	42.11
500	0.5614	94.44	5.3337	63.46	5.6145	47.37
1000	0.2807	97.22	3.9301	73.08	5.0530	52.63

3.3 Activation energy

The activation energy (E_a) for the corrosion of API – 5L – Grade – X60 Steel in the presence and absence of ESBL extract is calculated using the following Arrhenius equation (4) and its derived form equation (5).

CR= Aexp ($-E_a/RT$) -------------- (4)

log (CR_2/CR_1) = $E_a/2.303$ R ($1/T_1-1/T_2$) ------------- (5)

(Where CR_1 and CR_2 are the corrosion rate at the temperature T_1 and T_2 respectively, E_a is the activation energy and R is the universal gas constant).

The value of activation energy (E_a) for the blank (4.0183kJ/mol) is lower than in the presence of inhibitors (Table-3). This observation clearly indicates that the adsorption process also is physisorption.

3.4 Heat of adsorption

The heat of adsorption on the surface of various metals in the presence of plant extract in 1.0N HCl environment is calculated by the equation (6).

Q_{ads} =2.303 R [log (θ_2/1- θ_2)-log (θ_1 /1- θ_1)] x (T_2T_1/T_2-T_1) ------(6)

(Where R is the gas constant, θ_1 and θ_2 are the degree of surface coverage at temperatures T_1 and T_2 respectively).

The calculated Q_{ads} values (Table-3) are ranged from -104.9678 to -68.9052 kJ/mol. This negative value indicate and suggests that the adsorption of ESBL extract on the API – 5L – Grade – X60 Steel surface is exothermic.

Table 3: Calculated values of Activation energy (E_a) and heat of adsorption (Q_{ads}) of ESBL extract on API – 5L – Grade – X60 Steel in 1.0N HCl environment.

S.No	Conc. of inhibitor (ppm)	% of I.E		E_a ($KJmol^{-1}$)	Q_{ads} ($KJmol^{-1}$)
		30^o	60^o		
1.	0	-	-	4.0183	--
2.	10	88.89	15.79	46.7902	-104.9678
3.	50	91.67	36.84	58.1554	-82.1453
4.	100	94.44	42.11	67.0648	-88.1085
5.	500	94.44	47.37	64.3999	-82.1518
6.	1000	97.22	52.63	80.8385	-68.9052

3.5 Adsorption studies

Process of adsorption are very important phenomeno to determine the corrosion rate of reaction mechanism. The most frequently use of isotherms are viz: Langmuir, Temkin, Frumkin, Flory- Huggins, Freund lich, Bockris-Swinkles, Hill-de Boer, Parson's and the El-Amady, thermodynamic-kinetic model.

3.5.1. Langmuir, El-Awady and Temkin Adsorption Isotherm

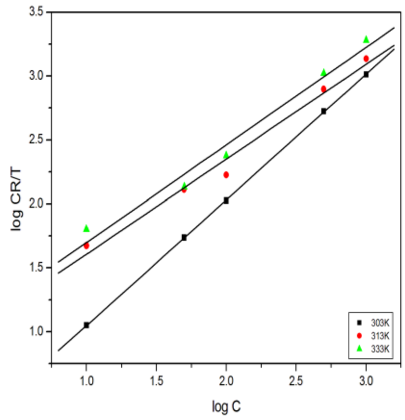
The Langmuir adsorption isotherm of ESBL extract on API – 5L – Grade – X60 Steel surface proceeded according to the following equation (7).

log C/θ = log C – log K --------(7)

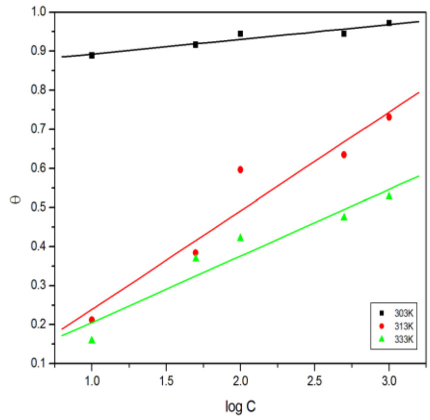
(Where θ is the degree of surface coverage, C is the concentration of the inhibitor solution and K is the equilibrium constant of adsorption of inhibitor on the metal surface).

By plotting the values of log(C/θ) Vs log C, linear plots are generated (fig-3). Inspection of this figure reveals that the experimental data fitted with the Langmuir adsorption isotherm, means that there is no interaction between the adsorbed species.

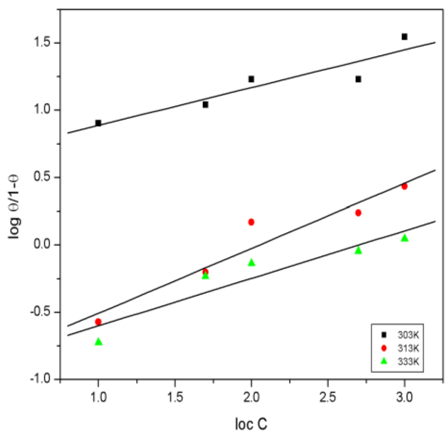
The Figs 2(a-c) shows Langmuir, El-Awady, and Temkin isotherm model respectively. The values of K, R^2 and ΔG_{ads} are derived from these adsorption isotherm at different temperature ranges from 303K to 333K are given in Table -4. The regression co-efficient values viz, El-Awady (R^2=0.9400) and Temkin (R^2=0.9551) Freundlich (R^2=0.9312) and Florry-Huggins (R^2=0.9315) adsorption isotherm models and this observed values relatively far from unity as compared with the values obtained with Langmuir adsorption isotherm (R^2=0.9937) which is clearly indicate that the average value of regression co-efficient is almost unity. Thus it is that the inhibitor obeys Langmuir adsorption isotherm.



(a)



(b)



(c)

Fig. 2: (a) Langmuir, (b) Temkin, (c) El-Awady isothserm for the adsorption of ESBL inhibitor on API – 5L – Grade – X60 Steel in 1.0N HCl.

3.5.2. Free energy of adsorption

The equilibrium constant of adsorption for various plant extract on the surface of API – 5L – Grade – X60 Steel is related to the free energy of adsorption ΔG_{ads} by equation (8).

ΔG_{ads} = -2.303 RT log (55.5 K) --------------------------------- (8)

(Where R is the gas constant, T is the temperature, K is the equilibrium constant of adsorption).

The values of intercept (K) obtained from Langmuir, El-Awady, Temkin adsorption isotherm is substituted in equation (8) and the calculated values of ΔG_{ads} are placed in Table-4. In El-Awady adsorption isotherm, the decrease of equilibrium constant (K_{ads}) values suggest that the process is physisorption phenomenon, which attributed to electrostatic interaction between the charged metal and active inhibitor molecules. Also the values of 1/y are less than unity, showing that there is a multilayer adsorption of the inhibitor molecule on the metal surface.[40,41] In Langmuir adsorption, the negative values of ΔG_{ads} suggested that the adsorption of ESBL extract onto API – 5L – Grade – X60 Steel surface is a spontaneous process and the adsorbed layer is more stable one.

Table 4: Langmuir, El-Awady and Temkin adsorption parameters for the adsorption of ESBL inhibitor on API – 5L – Grade – X60 Steel in 1.0N HCl.

Temperature (Kelvin)	Adsorption Isotherms									
	Langmuir			El-Awady				Temkin		
	K	R^2	$-\Delta G_{ads}$ kJ/mol	K	1/y	R^2	$-\Delta G_{ads}$ kJ/mol	K	R^2	$-\Delta G_{ads}$ kJ/mol
303K	1.1660	0.9999	-10.5066	4.0457	0.2802	0.9266	-13.6411	7.1597	0.9507	-15.0794
313K	7.3349	0.9918	-15.6399	0.1024	0.4824	0.9613	-4.5223	0.9689	0.9596	-10.3714
333K	8.6079	0.9895	-17.0825	0.1112	0.3528	0.9321	-18.1932	1.0851	0.9552	-11.3477

3.5.3 Thermodynamics parameters

An alternative formula of the Arrhenius equation is the transition state equation,

CR=RT/Nh exp (ΔS/R) exp (-ΔH/RT) ----- (9)

(Where h is the Planck's constant, N the Avogadro's number, ΔS the entropy of activation, and ΔH the enthalpy of activation).

A plot of log (CR/T) Vs. 1000/T gives a straight line (Fig. 3) with a slope of (−ΔH/R) and an intercept of [log(R/Nh)) + (ΔS/R)], from which the values of ΔS and ΔH were calculated and listed out in Table-5. The positive value of enthalpy of activation clear that the endothermic nature of dissolution process is very difficult. The positive value of entropy (ΔS) is gradually increasing at the solid/liquid interface during the adsorption of the metal ions onto inhibitor molecules present in the extract.

Table 5: Thermodynamic parameters of API – 5L – Grade – X60 Steel in 1.0N HCl obtained from weight loss measurements.

S.No	Concentration of CRL (ppm)	ΔH ($kJ\ mol^{-1}$)	ΔS ($J\ k^{-1} mol^{-1}$)
1	0	-0.1280	68.2699
2	10	15.2063	113.0849
3	50	20.3817	128.9680
4	100	24.5512	141.064
5	500	23.5369	137.6237
6	1000	30.2696	157.6097

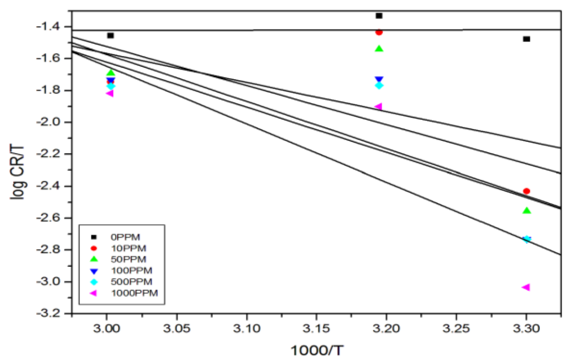


Figure 3: The relation between log (CR/T) and 1000/T for different concentrations of ESBL extract on API – 5L – Grade – X60 Steel.

4. CONCLUSIONS

Eugenia Singampattiana Beddome has shown excellent inhibition performance for API – 5L – Grade – X60 Steel in Crude oil with 10%, Free water environment. The inhibition efficiency increased with the increase of inhibitor concentration. The maximum inhibition efficiency was achieved 99.17%. Inhibition efficiency gradually decreased with the rise in temperature ie, 97.22% to 52.63% for 303K and 333K respectively. It follows physical adsorption mechanism. The value of activation energy (E_a), enthalpy of adsorption (ΔH_{ads}) and free energy changes (ΔG_{ads}) indicates that the adsorption of inhibitor on metal surface follows physical, exothermic and spontaneous process respectively. The inhibitor is found to obey Langmuir adsorption isotherms.

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