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# Data and statistical evaluation of the protection effect of essential oil extracts on plain carbon steel in dilute acid media

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**Abstract.** Optimal performance of corrosion inhibiting compounds significantly depends on specific range of inhibitor concentration and measurement time. Data on the protection effect of laurus cassia and rosmarinus angustifolius essential oil extracts on plain carbon steel in 0.5 M H<sub>2</sub>SO<sub>4</sub> and HCl electrolyte was researched into by weight loss analysis. The data showed the admixed extracts were highly potent at the concentrations studied with protection effect typically above 95%. The protection effect increased progressively with measurement time at 1% to 2% extracts concentration beyond which (2.5% to 3% concentration) the results were generally constant. The extracts exhibited inhibition performance independent of concentration. Data on standard deviation showed the degree of deviation of protection effect with measurement time and concentration is minimal. The fraction of protection effect data above 90% protection in H<sub>2</sub>SO<sub>4</sub> is 98% and 100% in HCl solution at limit of errors of 3.24% and 0%. Statistical data by analysis of variance shows both inhibitor concentration and measurement time remarkably altered the protection effect of the extracts with statistical relevance values of 47.17% and 53.02 for concentration and 26.51% and 29.52% in H<sub>2</sub>SO<sub>4</sub> and HCl solution.

## 1. Introduction

Carbon steel exhibits unique mechanical and physical properties which are important benchmarks against which the properties of other ferrous alloys can be correlated. The steel is the most versatile engineering alloy with applications worldwide [1]. However, the extensive application of carbon steels exposes them to corrosive anions especially in industries such as oil and gas production, power plants, energy generation, chemical processing, mining industry, due to the aqueous operating condition the steels are subjected to therein [2]. Corrosion of metallic alloys occurs due to the cathodic-anodic reaction mechanism on the surface. This mechanism is a function of the heterogeneity of the alloys, impurities and alloying elements [3]. The poor resilience of carbon steels to general corrosion, and in specific environments to localized deterioration is due the absence of passive film formation on its surface [4]. As a result, the operating lifespan of the steel is severely shortened. In most cases the lifespan of the steel significantly depends on the concentration of corrosive species in the aqueous environment. The consequences of this phenomenon are perpetual collapse of the steel, structural damage and continuous repairs at unscheduled downtimes which leads to avoidable accidents, fluid leakages and cost transfer to consumers/end-users. The most cost effect and long-lasting means of corrosion protection stems from the application of corrosion inhibitors. These are chemical derivatives which significantly suppress the electrochemical processes responsible for corrosion [5]. Conventional corrosion tends to be harmful to



personnel and environment [6]. Organic compounds prove to be effective corrosion inhibiting compounds are also noxious [7]. Plant extracts are promising compounds for corrosion inhibition of carbon steels. However, current research gives mixed results due to limitation in lifespan, poor adsorption behaviour etc. [8-12]. Oils extracts of plants with significant number of phytochemical compounds undergo numerous investigations to evaluate their protection effect properties with the results being appreciable [13-20]. However, evaluation of data on the inhibition of oil extracts is necessary to differentiate the optimal reaction of the extracts with regards to measurement time and concentration as most compounds are concentration dependent. This investigation focuses on data and statistical assessment of the protection effect of *Laurus cassia* and *rosmarinus angustifolius* essential oil extracts on plain carbon steel in 0.5 M  $\text{H}_2\text{SO}_4$  and HCl electrolyte.

## 2. Experimental methods

*Laurus cassia* and *rosmarinus angustifolius* essential oil (LCRA) procured from NOW foods in the United States of America in the synthesized form were prepared in volumetric quantities of 1%, 1.5%, 2%, 2.5% and 3% in 200ml of 0.5 M  $\text{H}_2\text{SO}_4$  and HCl electrolyte. Plain carbon steel (PCS) circular rods were cut with hacksaw into 6 pieces for weight loss measurement. Weight measured PCS samples were suspended in both acids within a beaker for 480 h. PCS pieces were measured every 1440 mins with Ohaus scale instrument. The weight loss was enumerated from the deduction between the initial weight of PCS (kept constant for 480 h) and sequent weights. Corrosion rate and inhibition efficiency were determined from the weight loss values.

Dual-component unitary component experimental ANOVA test (F - test) was employed to assess the statistical importance of LCRA concentrations and measurement time on LCRA protection output. Analysis was done at confidence level of 95% i.e. a significance level of  $\alpha = 0.05$  to determine the summation of squares of columns (measurement time), summation of squares among rows (oil extract concentration) and total computation of squares.

## 3. Results and discussion

### 3.1. Coupon measurement

Table 1 depict the corrosion rate values for PCS in  $\text{H}_2\text{SO}_4$  and HCl electrolyte at set LCRA concentrations while Table 2 depict the analogous protection effect of LCRA concentrations for 240 h. General examination of Table 1 affirms the corrosion rate values of PCS in  $\text{H}_2\text{SO}_4$  and HCl electrolyte at 0% LCRA is considerably higher than the results retrieved at the set LCRA concentrations. Secondly, the corrosion rate of PCS gotten from  $\text{H}_2\text{SO}_4$  solution is substantially more than the values obtained in HCl. The higher corrosion rate value in the absence of LCRA extract in both acids is due to accelerated oxidation of PCS exterior amidst highly reactive corrosion anions ( $\text{SO}_4^{2-}$  and  $\text{Cl}^-$ ).

$\text{H}_2\text{SO}_4$  in  $\text{H}_2\text{O}$  is highly reactive on PCS in aqueous environments inducing the ejection of  $\text{Fe}^{2+}$  ions into the acid. Consequently, the steel deteriorates analogous to exposure time in Table 1. Between 24 h and 144 h, the corrosion rates of PCS were relatively greater than the initial value (72.34 mm/y) in  $\text{H}_2\text{SO}_4$  due to increased ionization of the steel surface. Beyond 144 h, reduction in corrosion rate value was observed till 240 h (48.99 mm/y) because of dilution of the acid electrolyte with released corrosion particles. In HCl solution, the subsequent corrosion rate results were higher than the initial value of 16.45 mm/y (24 h) due to the localized reaction mechanism of  $\text{Cl}^-$  compared to  $\text{SO}_4^{2-}$  which is more aggressive and tends to induced general corrosion over the steel exterior. The greater corrosion rate outputs of PCS in  $\text{H}_2\text{SO}_4$  contrasted to HCl is because of the higher ionization constant of  $\text{H}_2\text{SO}_4$  in  $\text{H}_2\text{O}$  such that two H atoms are released as against HCl which releases only one hence  $\text{SO}_4^{2-}$  is more aggressive and reacts over the entire steel exterior. Substantial decrease in corrosion rate values of PCS was ascertained at specific LCRA concentrations. This is due to the protection effect of protonated LCRA compounds in the acid solution whereby the emission of corrosive species unto the steel exterior was hindered and the subsequent redox electrochemical process was suppressed. Corrosion rate data at

1% to 2.5% LCRA concentration within  $H_2SO_4$  electrolyte decreased with measurement time signifying time dependent effective inhibition action. However, at 3% LCRA concentration, increase in corrosion rate was observed with reference to measurement time despite substantial reduction in corrosion rate in comparison to the results at 0% LCRA concentration. This occurrence is results from lateral interaction effect among inhibitor molecules which tends to influence the overall performance of inhibitors. In HCl solution, reduction in corrosion rate values analogous to exposure time was observed from 1% to 2% LCRA concentration. At 2.5% LCRA concentration, increase in corrosion rate was observed for reasons earlier discussed while at 3% LCRA concentration, the corrosion rate results of PCS were generally constant with respect to exposure time. In both acid solutions, the performance of LCRA on PCS tends to be non-concentration dependent.

The corresponding protection effect results in Table 2 shows LCRA proves to be an effective inhibitor compound. General observation shows the average protection effect in both acids is above 95%. At 1% LCRA concentration in  $H_2SO_4$ , significant improvement in protection effect results was ascertained. The protection effect at 24 h is 88.4% while at 240 h it is 97.24%. Similar phenomenon occurred at 1.5% and 2% LCRA concentration. However, from 2.5% to 3% LCRA concentration the protection effect values were generally constant due to effective inhibition action and thermodynamic stability of the protective film. In HCl solution, increase in protection effect was observed from 24 h to 240 h of exposure. At 2.5% LCRA, the protection effect data were generally stable while at 3% LCRA concentration, the data changes with measurement time. The final protection effect data at all LCRA concentration shows the protection performance of LCRA is independent of its concentration. Comparison of the plot of protection effect versus measurement time at 1% and 3% LCRA concentration in  $H_2SO_4$  and HCl solution is depicted in Fig. 1(a) and (b). The plots in Fig. 1(a) depicts the performance of LCRA increases with exposure time before attained relative equilibrium at 120 h till 240 h at 1% LCRA concentration. At higher concentration of 3% the plot was generally stable throughout the exposure hours. The plot configuration of LCRA analogous to exposure time in HCl solution at 1% and 3% LCRA concentration [Fig.1(b)] significantly contrast the configuration in Fig. 1(a). It reveals the protection effect of LCRA on PCS in HCl is unstable and decreases analogous to measurement time.

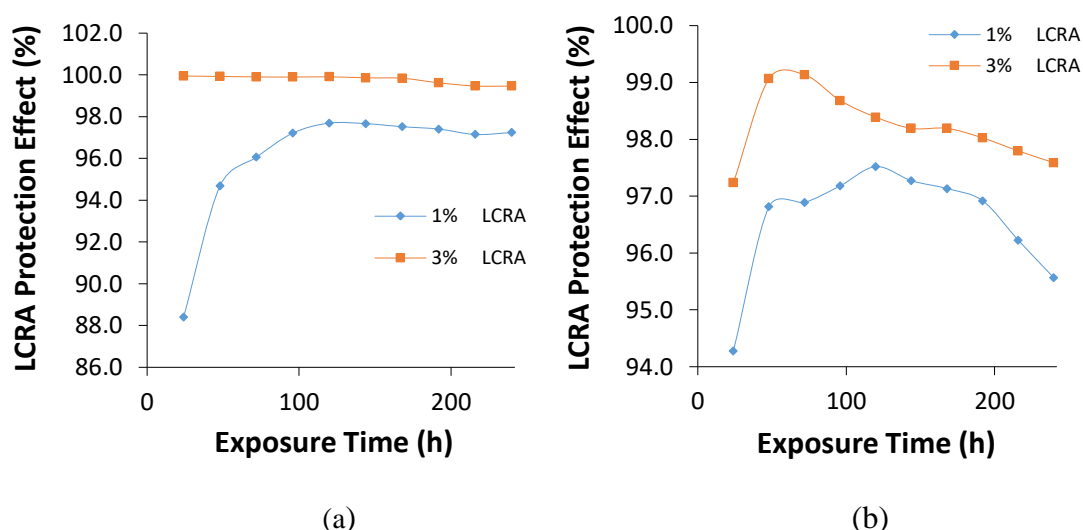


Fig. 1. Plot of LCRA protection versus exposure time at 1% and 3% LCRA concentration in (a)  $H_2SO_4$  and (b) HCl solution

**Table 1.** Corrosion rate data for LCS in H<sub>2</sub>SO<sub>4</sub> and HCl solution at specific LCRA concentrations.

LCRA Conc.	H <sub>2</sub> SO <sub>4</sub>						HCl					
	0% LCRA	1% LCRA	1.5% LCRA	2% LCRA	2.5% LCRA	3% LCRA	0% LCRA	1% LCRA	1.5% LCRA	2% LCRA	2.5% LCRA	3% LCRA
Exp. Time (h)												
24	72.34	8.39	3.24	4.09	0.53	0.04	16.45	0.94	0.96	0.61	0.10	0.45
48	89.84	4.77	1.65	2.18	0.30	0.06	31.68	1.01	0.31	0.31	0.18	0.30
72	81.02	3.19	1.27	1.59	0.21	0.08	29.02	0.90	0.23	0.20	0.13	0.25
96	88.70	2.47	1.06	1.31	0.18	0.09	29.65	0.84	0.18	0.16	0.11	0.39
120	87.81	2.02	0.86	1.17	0.16	0.08	28.68	0.71	0.14	0.13	0.09	0.46
144	73.65	1.72	0.43	0.60	0.15	0.10	26.09	0.71	0.13	0.11	0.10	0.47
168	63.54	1.58	0.86	1.04	0.15	0.10	24.16	0.69	0.13	0.10	0.10	0.44
192	56.65	1.47	0.80	1.00	0.20	0.21	22.96	0.71	0.14	0.09	0.15	0.45
216	52.12	1.49	0.80	1.09	0.32	0.28	20.48	0.77	0.13	0.11	0.15	0.45
240	48.99	1.35	0.74	1.07	0.35	0.26	18.54	0.82	0.22	0.12	0.15	0.45

**Table 2.** Protection effect data for LCRA on PCS in H<sub>2</sub>SO<sub>4</sub> and HCl solution at specific LCRA concentrations.

LCRA Conc.	H <sub>2</sub> SO <sub>4</sub>					HCl				
	1% LCRA	1.5% LCRA	2% LCRA	2.5% LCRA	3% LCRA	1% LCRA	1.5% LCRA	2% LCRA	2.5% LCRA	3% LCRA
Exp. Time (h)										
24	88.40	95.52	94.35	99.26	99.95	94.28	94.16	96.29	99.41	97.24
48	94.69	98.16	97.57	99.67	99.93	96.81	99.03	99.04	99.44	99.07
72	96.06	98.43	98.03	99.74	99.90	96.89	99.22	99.30	99.56	99.14
96	97.22	98.81	98.52	99.80	99.90	97.18	99.41	99.46	99.64	98.68
120	97.69	99.02	98.67	99.82	99.91	97.52	99.51	99.55	99.67	98.39
144	97.66	99.42	99.18	99.79	99.86	97.27	99.51	99.58	99.62	98.19
168	97.52	98.65	98.37	99.77	99.84	97.13	99.47	99.60	99.58	98.19
192	97.41	98.59	98.24	99.65	99.62	96.92	99.37	99.63	99.34	98.02
216	97.15	98.46	97.91	99.39	99.47	96.23	99.35	99.49	99.29	97.80
240	97.24	98.48	97.82	99.28	99.46	95.56	98.79	99.38	99.21	97.59

### 3.2. Statistical analysis

Data on standard deviation, mean and limit of error for LCRA protection effect analogous to concentration in H<sub>2</sub>SO<sub>4</sub> and HCl solution are revealed in Table 3 and 4. The standard deviation values for LCRA in H<sub>2</sub>SO<sub>4</sub> solution varies with LCRA concentration. The highest standard deviation result of 2.86 was observed at 1% LCRA concentration while the lowest value occurred at 3% LCRA concentration. The value at 1% LCRA is due to the significant difference in protection effect data relative to the mean value. The low standard deviation values at 3% LCRA signifies minimal changes in protection effect data from average protection value due to stable protection of the steel over time. Stable inhibition performance is prevalent at 2.5% and 3% LCRA concentration. The standard deviation values for LCRA protection effect data in HCl shows LCRA concentration at 2.5% and 3% exhibited the most stable protection performance analogous to measurement time. Data on limit of error shows 98% and 100% of protection effect data in H<sub>2</sub>SO<sub>4</sub> and HCl electrolyte are greater than 90% inhibition performance at limit of error of 3.24% and 0% respectively.

Analysis of variance (ANOVA) was used to analyse the statistical importance of LCRA inhibitor concentration and measurement time on the protection effect of LCRA. The mean square ratio for the sources of variation in H<sub>2</sub>SO<sub>4</sub> and HCl solution are higher than the theoretical significance factor. Observation of the statistical relevance factor shows LCRA concentration and measurement time significantly influence the performance output of LCRA extract. However, in H<sub>2</sub>SO<sub>4</sub> and HCl solution LCRA concentration is has more influence with statistical relevance factor of 47.17% and 53.02% relative to the measurement time with values of 26.51% and 29.52%.

**Table 3.** Data on standard deviation, mean and limit of error for LCRA protection effect in H<sub>2</sub>SO<sub>4</sub> and HCl solution.

	H <sub>2</sub> SO <sub>4</sub>				HCl					
Conc. (%)	1	1.5	2	2.5	3	1	1.5	2	2.5	3
SD	2.86	1.05	1.32	0.22	0.19	0.99	1.64	1.01	0.16	0.61
Mean	96.10	98.35	97.87	99.62	99.78	96.58	98.78	99.13	99.48	98.23
Limit of Error	±3.24%		Data above 90% Protection Effect		98%	Limit of Error	±0%		Data above 90% Protection Effect	
										100%

**Table 4.** ANOVA data for statistical influence of LCRA concentration and exposure time on LCRA protection effect

	H <sub>2</sub> SO <sub>4</sub>			HCl			
Source of Variation	Mean Square Ratio (F)	Theoretical Significance Factor	Statistical Relevance Factor, F (%)	Source of Variation	Mean Square Ratio (F)	Theoretical Significance Factor	Statistical Relevance Factor, F (%)
CCRO Concentration Measurement	16.13	2.42	47.17	CCRO Concentration Measurement	27.32	2.42	53.02
Time	4.03	2.1	26.51	Time	6.76	2.1	29.52

#### 4. Conclusion

Extracts of laurus cassia and rosmarinus angustifolius adequately prevented the surface oxidation of plain carbon steel in dilute H<sub>2</sub>SO<sub>4</sub> and HCl electrolyte. Protection effect results changed substantially with measurement time at low to mid extract concentration. At higher extract concentration, protection effect data was predominantly stable analogous to measurement time. Inhibition performance of the extract varied slightly with concentration signifying concentration dependent action. Data on standard deviation shows protection effect of the extracts is thermodynamically stable with major proportion of the data being above 90% inhibition. Statistical data shows inhibitor concentration and measurement time are statistically important to the protection output of the extracts at varying degrees.

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