

Inhibition Performance of Admixed Grapefruit and Lemongrass Oil Extracts on Low Carbon Steel in Weak Acid Formulation



R. T. Loto, P. Okpaleke, and U. Udoh

1 Introduction

The universal utilization of carbon steels worldwide for structural components and platforms coupled with their inherent mechanical, physical, and metallurgical properties establishes them as the standard wherewith the properties of other metallic alloys can be compared (Dwivedi et al., 2017). Carbon steels are applied in construction, petrochemical crude distillation unit, mining, energy generation, automobile, and desalination plants. However, the weak resistance of the steel to corrosion severely shortens their operational life span because of the reaction effect of SO_4^{2-} , Cl^- , $\text{S}_2\text{O}_3^{2-}$, NO_3^- , etc. in aqueous environments on the steel surface (Zarras & Stenger-Smith, 2017; Loto & Babalola, 2017). The capacity of carbon steels to be inert is because of the absence of important passivating species that is responsible for their weak corrosion resistance. As a result, the cost of corrosion control, repair, replacement of damaged parts, and continual maintenance to prevent collapse of structure, industrial accidents, fluid leakages, etc. is shifted to consumers/end-users. Corrosion inhibitors are chemical derivatives which remain the most versatile and cost-effective method of controlling carbon steel degradation during service (Branko, 2015). The adequate corrosion inhibitors in service are inorganic and tend to be very poisonous (Singh & Bockris, 1996). Synthetic organic compounds which have seen limited service are also toxic to personnel and the environment (Brycki et al., 2017; Loto, 2017; Loto & Loto, 2012, 2013). Research on plant extracts for application as corrosion inhibitor is ongoing. The results gotten so far are appreciable; however, plant extracts are limited by short shelf life, weak adsorption, and general poor inhibition (Li et al., 2005; Quartarone et al., 2012; Ashassi-Sorkhabi et al., 2004; ÖZcan, 2008; Loto & Oghenerukewe, 2016). Essential oils extracts

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have phytochemical compounds with numerous research being done to assess their corrosion inhibition properties (Fu et al., 2010; Bouoidina et al., 2017; Hamdani et al., 2015; El Ouadi et al., 2014; El Ouariachi et al., 2015; Boumhara et al., 2015; Lahhit et al., 2011; Loto et al., 2018). Results have shown they tend to be effective at higher concentrations, and their performance can be strongly dependent on concentration and exposure time. Non-oil extract-based inhibitors have been proven to be effective. However, research on them is ongoing before commercialization (Ye et al., 2020a, b, 2021; Turcio-Ortega et al., 2007; Guadalupe et al., 2011; Hussin et al., 2016; Cruz et al., 2005). Synergistic combination effect of the oil extracts has given more effective results. However, data investigation of the inhibition output is important to establish the peak performance of the extracts with respect to experimental factors. This investigation targets the corrosion rate and performance output of admixed grapefruit and lemongrass oil extracts on low carbon steel in 0.5 M H₂SO₄ and HCl formulation, and statistical investigation of the data.

2 Experimental Methods

Grapefruit and lemongrass essential oils (GFLG) are produced by NOW Foods, United States, and obtained through an intermediary in the synthesized form with assured 100% purity which were admixed together in ratio 1:1 and formulated in volume concentrates of 1.0%, 2.0%, 3.0%, 4.0%, and 5.0% in 200ml of 0.5 M H₂SO₄ and HCl acid formulation. Low carbon steel (LCS) rods with diameter of 1.2 cm and average thickness of 1 cm have wt. % constituent of 0.06% S, 0.05% P, 0.9% Mn, and 98.99% Fe by confirmation with PhenomWorld scanning electron microscope. Low carbon steel (LCS) was prepared into six test subjects for coupon investigation. LCS samples were sandpapered with coarse SiC polishers (60–2500 grits) before burnishing with 6 μm Struers DiaDuo-2 polishing formulation and subsequently rinsed with acetone. Computed LCS samples were inserted into the acid formulation for 240 h. LCS samples were weighed at 24 h interludes with Ohaus scaling instrument. The weight difference was computed from the deduction between the starting weight of LCS (maintained for 240 h) and successive weight recorded at 24 h intermittently, amounting to 240 h in total. Corrosion rate of LCS was computed according to the equation below (Ali & Fulazzaky, 2020; Khaksar & Shirokoff, 2017):

$$R = \left[\frac{87.6W}{DAT} \right] \quad (1)$$

W represents weight loss (g), D represents density (g/cm³), A represents area (cm²), and T represents time of measurement (h). Inhibition output (η) was computed according to Eq. 2:

$$\eta = \left[\frac{\omega_1 - \omega_2}{\omega_1} \right] \times 100 \tag{2}$$

ω_1 represents weight loss of LCS free the plant extracts and ω_2 represents weight loss of LCS at set GL concentrations. Binary-part mono level ANOVA test (F-test) was utilized to investigate the statistical applicability of GFLG concentrations and exposure time on GFLG inhibition data. The assessment was executed at confidence factor of 95% (significance level of $\alpha = 0.05$) analogous to the following successive equations. The aggregation of squares of columns (exposure time) was computed as follows:

$$SS_c = \frac{\sum T_c^2}{nr} - \frac{T^2}{N} \tag{3}$$

The summation of squares between rows (extract concentrate) was computed with Eq. 4:

$$SS_r = \frac{\sum T_r^2}{nc} - \frac{T^2}{N} \tag{4}$$

Total computation of squares as:

$$SS_{Total} = \sum x^2 - \frac{T^2}{N} \tag{5}$$

3 Results and Discussion

3.1 Gravimetric Analysis

Table 1 presents the corrosion rate data for LCS from 0.5 M H₂SO₄ and HCl formulations, and Table 2 presents the analogous data for GFLG inhibition output from both acids. Inspection of Table 1 reveals the substantial variation in LCS corrosion rate from both acids at 0% GFLG and GFLG at 1–5% concentration. Figure 1 exhibits the comparative plots of GFLG inhibition output at 1% and 5% GFLG concentration. At 0% GFLG, the corrosion rate initiated at 102.24 and 62.071 mm/y (24 h) in H₂SO₄ and HCl formulations, and terminated at 34.59 and 17.53 mm/y (240 h). The higher corrosion rate is due to accelerated degradation of LCS due to SO₄²⁻ and Cl⁻ anions. The anions oxidize the steel surface causing the rapid discharge of ionized steel cations into the electrolyte. LCS corrosion rate value (0% GFLG) is significantly higher in H₂SO₄ than HCl by reason of the diprotic nature of H₂SO₄ when it ionizes in H₂O compared to HCl which is monoprotic. As a result,

Table 1 LCS corrosion rate data from H₂SO₄ and HCl solution after 240 h of exposure

Exp. time (h)	LCS corrosion rate (mm/y) in H ₂ SO ₄ solution						LCS corrosion rate (mm/y) in HCl solution					
	GFLG conc. (%)											
	0% GL	1.0% GL	2.0% GL	3.0% GL	4.0% GL	5.0% GL	0% GL	1.0% GL	2.0% GL	3.0% GL	4.0% GL	5.0% GL
24	102.24	0.662	0.818	0.006	0.253	0.500	62.071	0.247	0.337	0.350	0.136	0.247
48	59.75	0.370	0.464	0.013	0.286	0.471	31.626	0.136	0.204	0.237	0.107	0.211
72	59.00	0.264	0.355	0.095	0.201	0.405	32.799	0.229	0.164	0.171	0.102	0.147
96	44.58	0.140	0.112	0.086	0.154	0.451	24.263	0.219	0.138	0.217	0.088	0.117
120	50.75	0.312	0.104	0.092	0.129	0.476	27.640	0.276	0.148	0.266	0.104	0.104
144	42.66	0.344	0.217	0.093	0.124	0.449	23.943	0.242	0.275	0.328	0.111	0.114
168	46.13	0.351	0.205	0.160	0.134	0.452	23.820	0.271	0.286	0.345	0.107	0.211
192	40.97	0.340	0.204	0.153	0.187	0.467	20.895	0.273	0.288	0.449	0.196	0.312
216	37.24	0.402	0.221	0.163	0.216	0.427	19.358	0.387	0.485	0.479	0.248	0.344
240	34.59	0.376	0.221	0.172	0.220	0.466	17.530	0.474	0.541	0.646	0.282	0.399

Table 2 GFLG inhibition efficiency data from H₂SO₄ and HCl solution after 240 h of exposure

Exp. time (h)	GFLG inhibition efficiency (%) in H ₂ SO ₄					GFLG inhibition efficiency (%) in HCl				
	GFLG conc. (%)									
	1.0% GL	2.0% GL	3.0% GL	4.0% GL	5.0% GL	1.0% GL	2.0% GL	3.0% GL	4.0% GL	5.0% GL
24	99.35	99.20	99.99	99.75	99.51	99.60	99.46	99.44	99.78	99.60
48	99.38	99.22	99.98	99.52	99.21	99.57	99.35	99.25	99.66	99.33
72	99.55	99.40	99.84	99.66	99.31	99.30	99.50	99.48	99.69	99.55
96	99.69	99.75	99.81	99.65	98.99	99.10	99.43	99.10	99.64	99.52
120	99.39	99.80	99.82	99.75	99.06	99.00	99.46	99.04	99.62	99.62
144	99.19	99.49	99.78	99.71	98.95	98.99	98.85	98.63	99.53	99.53
168	99.24	99.56	99.65	99.71	99.02	98.86	98.80	98.55	99.55	99.11
192	99.17	99.50	99.63	99.54	98.86	98.70	98.62	97.85	99.06	98.51
216	98.92	99.41	99.56	99.42	98.85	98.00	97.49	97.53	98.72	98.22
240	98.91	99.36	99.50	99.36	98.65	97.29	96.91	96.31	98.39	97.72

degradation by SO₄²⁻ anions tends to be general over the steel exterior compared to degradation by Cl⁻ which tends to be localized at specific sites/regions of the steel (Loto et al., 2020). Moreover, LCS corrosion rate in both acids at 0% GFLG decreases significantly akin to exposure time by reason adulteration of the acid formulation with released corrosion precipitates. Nevertheless, the degradation rate of LCS at 0% GFLG remains significantly higher than values obtained higher GFLG concentration. The significant decrease in LCS corrosion from 1% to 5% GFLG concentration is because of the reaction effect of protonated GFLG molecules which obstructs the reduction-oxidation reaction processes inducing corrosion. As a result, discharge of Fe²⁺ cations from the steel exterior is effectively suppressed. The corrosion rate data for LCS at 1–5% GFLG concentration shows GFLG concentration

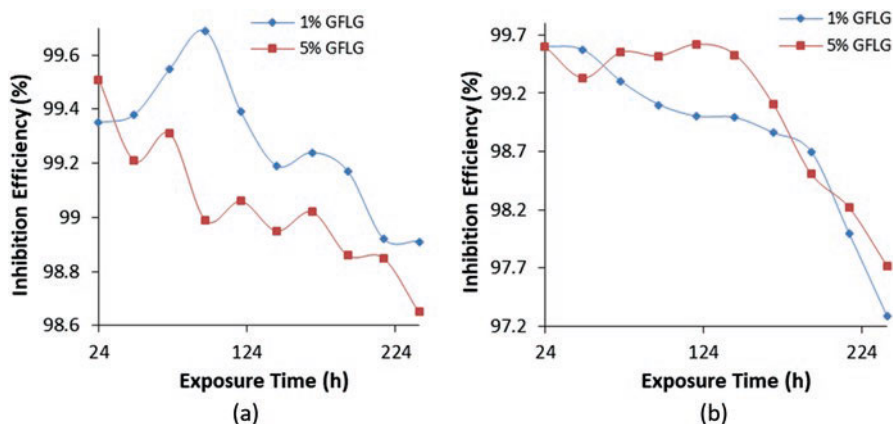


Fig. 1 Relative plot of GFLG inhibition output at 1% and 5% concentration in (a) H₂SO₄ formulation and (b) HCl formulation

has weak impact on the corrosion rate outputs of LCS. The rate of degradation of LCS in H₂SO₄ decreases analogous to exposure time, while in HCl the values increase. In H₂SO₄ solution, LCS corrosion rate values (1% and 5% GFLG concentration) initiated (24 h) at 0.662 and 0.5 mm/y. The correlating outcomes in HCl solution are 0.247 and 0.247 mm/y. At 240 h, the corrosion rate values in both acids declined to 0.376 and 0.466 mm/y in H₂SO₄, and 0.474 and 0.399 mm/y in HCl. These observations show GFLG improves in performance with time in H₂SO₄, while in HCl its performance marginally decreases with exposure time. Cl⁻ ions are significantly smaller than their SO₄²⁻ ion counterparts. As a result, limited transport of the Cl⁻ ion in the acid electrolyte diffuses through the barrier film formed by the oil extract on the steel (Cabrini et al., 2015; Loto, 2013). However, this observation does not negate the overall outstanding performance of the oil extract. Observation of inhibition efficiency data in Table 2 shows GFLG adequately inhibited the steel at all concentrates with protection performance values greater than 95% throughout the exposure hours. The inhibition output at 1% and 5% GFLG concentration in both acids at 24 h are 99.35% and 99.51% (H₂SO₄), and 99.60% and 99.60% (HCl). Comparing the data at 24 h to values at 240 h (98.91% and 98.65% in H₂SO₄) and (97.29% and 97.72% in HCl) shows the performance of GFLG as earlier stated is marginally independent of exposure time and concentration as presented in Fig. 1a, b. The trend fluctuation in Fig. 1a, b shows that inhibition output decreases with variation in exposure time. Moreover, the visible fluctuation in Fig. 1a compared to Fig. 1b showed the inhibition output of GFLG in H₂SO₄ is thermodynamically unstable compared to its performance in HCl. The statement has been added to the revised manuscript.

Data on standard deviation (SD), mean, and margin of error for GFLG inhibition values from H₂SO₄ and HCl formulation at set GFLG concentration are depicted in Table 3. The standard deviation data for GFLG in both acids generally vary with concentration. In H₂SO₄ the SD values varies from 0.14 at 4% concentration to 0.25

Table 3 Data on SD, mean, and margin of error for GFLG inhibition efficiency in H₂SO₄ and HCl solution

	H ₂ SO ₄					HCl				
GFLG Conc. (%)	1	2	3	4	5	1	2	3	4	5
SD	0.25	0.20	0.17	0.14	0.25	0.71	0.91	1.01	0.48	0.68
Mean	99.28	99.47	99.76	99.61	99.04	98.84	98.79	98.52	99.36	99.07
Margin of error	±0%	±0%	Result above 98% inhibition	100%	Margin of error	±0.81%	Result above 98% inhibition	88%	Margin of error	±0%

Table 4 ANOVA data for statistical influence of GFLG concentration and exposure time on GFLG inhibition performance

H ₂ SO ₄				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 (%)
Inhibitor conc.	30.86	2.42	62.38	Inhibitor conc.	15.20	2.42	12.90
Exposure time	4.27	2.1	19.43	Exposure time	41.60	2.1	79.46

at 1% and 5% concentrations. These values show the amount of deviation of GFLG inhibition data from mean value is significantly limited, i.e., it is generally stable analogous to exposure time. However, it tends to be more stable at 4% GFLG concentration. 4% GFLG concentration represents the limit of stability for GFLG performance on LCS in H₂SO₄ solution. Beyond 4% GFLG concentration, the SD values increase to 0.25 due to lateral interaction effect among GFLG ionic species in the electrolyte. Data shows the mean value varies with the 99% range due to steadiness of the inhibitor action upon the steel exterior. The SD data from HCl formulation are generally more than the data obtained in H₂SO₄ solution. The SD values vary from 0.48 at 4% GFLG concentration to 1.01 at 3% concentration. These values show the inhibition effect of GFLG in HCl is less steady akin to exposure time and its performance in H₂SO₄. The inhibition efficiency data in HCl is determined to vary relatively significantly with exposure time signifying increased or decreased performance over time. However, the mean values confirm effective performance. Data on margin of error depicts 100% and 88% of inhibition output in H₂SO₄ and HCl solution are above 98% inhibition performance at margin of error of ±0% and ±0.81%, respectively.

The effect of GFLG concentration and measurement time (sources of variation) on LCS corrosion was assessed with analysis of variance (ANOVA) and the data is laid out in Table 4. The statistical relevance component depicts the percentage degree of influence of the origin of variation on the performance output of

GFLG. Table 4 shows inhibitor concentration dominates the inhibition effect of GFLG on LCS at 62.38% compared to exposure time at 19.43%. The comparable values for HCl reveal exposure time dominates the performance of GFLG in HCl at 79.46% compared to GFLG concentration at 12.9%. The mean square ratio depicts the statistical data which must be more than the theoretical significance factor for the statistical relevance factor to be important. The mean square ratio for the origin of variation in H₂SO₄ and HCl formulation presented in Table 4 is more than the theoretical significance factor. Hence, the statistical relevant factors are valid.

4 Summary

Data assessment of the protection effect of admixed grapefruit and lemongrass oil extract on low carbon steel in dilute acid media was performed. Results showed the distillates adequately inhibited at all concentrations with average inhibition effect of 95%. Inhibition output from H₂SO₄ solution was determined to be significantly subject to concentration compared to the observation from HCl solution which was determined to be dependent on exposure time. The extracts' performance was determined to be more stable in H₂SO₄ solution compared to HCl with respect to exposure time. At 98% inhibition efficiency threshold, 100% of the extracts' inhibition on low carbon steel in H₂SO₄ were above the threshold, while 88% of the values in HCl were above the threshold.

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