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Corrosion inhibition of grapeseed oil on high carbon steel and ferrovanadium alloy in H_2SO_4 solution: Experimental and statistical analysis

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Abstract. Experimental and statistical analysis of the corrosion inhibition properties of grapeseed oil (GSO) on high carbon steel (HCS) and ferrovanadium (FVA) in 0.25 M H_2SO_4 solution was done. Results from gravimetric analysis show GSO performed more effectively on HCS with average inhibition efficiency above 90% at all concentrations compared to FVA where the values where generally around 70% at 360 h of exposure. Inhibition efficiency results did not vary significantly with GSO concentration compared to exposure time over 360 h. Secondly, inhibition efficiency values increased with respect to exposure time from initiation. ANOVA statistical method at 95% confidence level and significance level of 0.05 shows the sources of data variation (exposure time and inhibitor concentration) has minimal influence on the inhibition performance output of GSO. This indicate they are statistically indeterminate. The inhibition data is due to factors relating to presence of phytochemical properties, organo-metallic interaction, adsorption mode and influence of corrosive species. Data from standard deviation shows GSO inhibition efficiency data varies minimally from mean inhibition values from HCS which contrast the values obtained for FVA which were significantly high. The results show greater thermodynamic stability of GSO inhibition on HCS compared to FVA. 82% of GSO inhibition data from its action on HCS are above 70% inhibition compared to 22% for FVA.

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1 Introduction

Corrosion of metallic alloys from contact with acidic, alkaline, and saline solutions in industrial applications is responsible for the degradation and structural damage of the alloys, inevitably resulting in significant financial consequences [1-4]. The most important structural alloys of industrial significance are carbon steels and other low-cost ferrous alloys. Ferrous alloys are extensively applied in various engineering, manufacturing and construction applications due to its adaptable mechanical and physical properties. Despite these qualities ferrous alloys have very weak corrosion resistance [5-7]. Corrosion of ferrous alloys can be significantly reduced or prevented with the application of chemical compounds known as corrosion inhibitors. Corrosion inhibitors are considered one of the most important long-term solution to protect and extend the lifespan of ferrous alloys. These compounds are especially important in industrial processes such as the acid pickling process and oil-well acidizing due to the huge application of acids such H_2SO_4, HCl, HNO_3 etc. [8]. Corrosion inhibitors are categorized with respect to their reactivity, inhibition mode, toxicity etc. Their effectiveness and demand, centers around their ease of application, availability, affordability and performance [9-10]. A large category of effective corrosion inhibitors is of organic and inorganic origin. Organic corrosion inhibitors are generally toxic to the environment and personnel handling them. These compounds contain electronegative atoms, unsaturated bonds and the plane conjugated systems [11-19]. Application of organic inhibitors are increasingly limited by evermore stringent environmental regulations and adherence to United Nations Sustainable Development Goals. Environmentally friendly corrosion inhibitors are non-toxic and biodegradable compounds. Previous research have shown that these compounds inhibit the corrosion of metals in corrosive environments [20-28]. As part of the ongoing effort on the documentation of the performance of green chemical compounds for corrosion inhibition this article focusses on the inhibition effect of grapeseed essential on high carbon steel and ferrovanadium alloy in dilute H_2SO_4 solution. High carbon steel has seen extensive application as cutting tools, springs, washers, fasteners and coils in automotive, ship building, construction, petrochemical, chemical processing and mining industries. Ferrovanadium is applied as an additive to enhance the quality of ferroalloys. It is used to produce hand tools, gear components, crankshafts, axles and other critical steel products in construction, architecture and infrastructure projects. Ferrovanadium is also applied in the chemical processing industry due to its resistance to corrosive fluids

2 Material and methods

2.1 Sample preparation and weight loss analysis

304 Oil extracts from grapeseed (GSO)were added into a 0.25 M H_2SO_4 solution at concentrations of 0.5%, 1%, 1.5%, 2%, 2.5% and 3%. High carbon steel (HCS) and ferrovanadium alloy (FV) were divided into five test pieces for weight loss analysis. The test pieces were cleaned with distilled H_2O and C_3H_6O before the analysis. The HCS and FV test pieces were then submerged in the acid/extract solution for 480 h. The weight of the test pieces was documented every 24 h using a digital weighing instrument (resolution = 0.0001g, optimal capacity = 110g). Weight loss was computed by deducting the initial weight of HCS and FV from the subsequent weights measured at 24-hour intervals for a total of 480 h. The corrosion rate of HCS and FV was estimated using the equation below [23];

$$C_R = \left[\frac{87.6W}{\rho AT}\right] \quad (1)$$

W represents weight loss (g), ρ represents density (g/cm²), A represents area (cm²), and T represents the time of exposure (h). The inhibition efficiency (η) was calculated from equation 2 [23].

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

ω_1 represents weight loss of HCS and FV from the acid solution without the extracts while ω_2 denotes weight loss of HCS and FV at precise GSO concentration.

2.2 Statistical computation

A two-way ANOVA test (F-test) was employed to determine the statistical significance of GSO concentrations and exposure time on the inhibition performance results of GSO. The analysis was conducted at a 95% confidence level, meaning a significance level of α = 0.05, according to the numerical expressions below. The sum of squares for the columns (measurement time) was calculated from equation 3.

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

The combination of squares between the rows (GSO concentration) was gotten from equation 4 [24].

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

The total combination of squares is given in equation 5.

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

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Table 1 Elemental %Wt. composition of Al 4032, Mg-Ti, Al 4004 and Al-V

Elemental Composition (%)	Fe	Si	C	Cr	Mn	P	S	Ni	Al	N	Mo	Zn	Ca	Mg	Ti+Nb
X77ST	77.85	0.31	-	5	1.02	-	-	-	0.02	-	-	10.65	2.02	4.2	-
F20ST	79.34	0.1	0.02	20	0.2	0.03	0.003	-	-	0	-	-	-	-	0.3

304ST	70.75	1	0.08	18	2	0.045	0.03	8	-	0.1	0	-	-	-	-

3 Results and discussion

3.1 Corrosion rate studies

Table 2 shows the inhibition efficiency results for HCS and FVA in 0.25 M H_2SO_4 solution at specific concentrations of GSO (0.5% to 3% GSO) while Table 3 shows the corresponding corrosion rate results. GSO generally performed more effectively on HCS compared to FVA from observation of GSO inhibition efficiency results at 360 h of exposure. However, GSO performance with respect to time on both alloys varies. At 24 h, performance of GSO was significantly below the threshold for effective inhibition on both alloys. On HCS, GSO performance varies between 31.52% and 56.75%. There is a general increase in inhibition efficiency after 24 h of exposure. However, significant results were obtained at 192 h of exposure on HCS where the inhibition efficiency data varies from 60.88% at 1.5% GSO concentration to 74.45% at 2.5% GSO concentration. At 216 h of exposure, effective inhibition of HCS was attained at all GSO concentration with values generally above 80% inhibition efficiency. This trend continues to 360 h of exposure where above 90% inhibition was attained at all GSO concentrations. This trend contrast what was observed for GSO performance on FVA where the inhibitor generally performed poorly before 264 h of exposure. At 24 h, inhibition efficiency varies from 9.31% at 0.5% GSO to 44.96% at 1% GSO concentration. At 264 h of exposure, inhibition efficiency varies from 60.59% at 3% to 67.92% at 1% GSO concentration. Whereas at 360 h of exposure, the final corrosion rate varies from 70.13% at 3% GSO to 73.95% at 2% GSO concentration. The final inhibition efficiency results at 360 h of exposure shows GSO performance, while been dependent on exposure time due to progressive increase in corrosion rate does not shows centration dependent behavior.

The corresponding corrosion rate results are directly proportional to the inhibition efficiency results but allows for comparative analysis with HCS and FVA at 0% GSO concentration. At 24 h of exposure, corrosion rate of HCS and FVA at 0% GSO concentration are significantly higher than the corresponding corrosion rate values at 0.5% to 3% GSO concentration. This is due to the electrochemical action of SO_4^{2-} anions from the acid solution which oxidizes HCS and FVA surfaces causes the release of Fe^{2+} ions into the acid, invariably causing accelerated corrosion in the absence of GSO concentration. The corrosion rate at 24 h for HCS and FVA at 0% GSO are 22.76 mm/y and 125.22 mm/y. With respect to exposure time, corrosion rate of both alloys decreased to 14.23 mm/y and 33.60 mm/y. These values show that FVA is more susceptible to corrosion than HCS. In the presence of GSO compound, corrosion rate of HCS at 24 h varied from 9.84 mm/y at 3% GSO to 13.11 mm/y at 3% GSO concentration. The corresponding values for FVA at 24 h are 98.97 mm/y at 3% GSO concentration to 113.56 mm/y at 0.5% GSO concentration. Corrosion rate values for FVA at 24 h are significantly higher than the values for HCS. Observation shows that corrosion rate values for HCS decreased with respect to exposure time to values between 0.71 mm/y and 1.16 mm/y at all GSO concentrations. While the values for FVA at 360 h of exposure indicates are significantly higher from 8.79 mm/y to 10.04 mm/y.

Table 2 Inhibition efficiency data for HCS and FVA in 0.25 M H_2SO_4 solution at 0% to 3% GSO concentration

HCS						
GSO Conc. (%) / Exp. Time (h)	**0.5% GSO**	**1% GSO**	**1.5% GSO**	**2% GSO**	**2.5% GSO**	**3% GSO**
24	42.38	31.52	48.81	45.14	50.47	56.75
48	43.98	23.76	52.68	44.59	49.59	45.92
72	43.80	26.05	49.77	50.57	53.42	43.37
96	46.87	28.20	48.44	46.52	51.90	43.06
120	48.61	32.29	43.28	48.67	54.58	46.28
144	68.91	59.10	55.89	69.70	72.16	66.20
168	72.22	63.33	59.46	73.23	74.88	68.22
192	73.38	63.89	60.88	73.36	74.45	67.99
216	89.64	86.16	84.81	89.24	89.59	87.18
240	86.14	81.27	79.67	86.01	86.19	83.12
264	91.01	88.32	86.69	90.50	91.27	88.96
288	91.68	89.17	86.82	90.15	91.70	88.65
312	93.35	90.21	89.43	92.25	93.00	90.82
336	94.74	92.22	91.58	94.05	94.54	92.95
360	95.00	92.20	91.85	93.96	94.84	93.01

FVA						
GSO Conc. (%) / Exposure Time (h)	0.5% GSO	1% GSO	1.5% GSO	2% GSO	2.5% GSO	3% GSO
24	9.31	44.96	43.99	43.35	44.83	20.96
48	-1.21	18.53	19.18	38.58	11.28	16.90
72	1.10	15.57	12.16	32.56	3.58	16.72
96	-0.17	14.84	8.07	22.63	2.30	14.29
120	0.80	14.21	7.56	19.89	1.93	12.06
144	21.65	31.22	24.41	34.49	20.70	25.62
168	26.40	35.31	28.63	37.07	24.57	26.71
192	27.88	35.44	28.59	37.09	25.14	26.57
216	48.18	53.68	48.47	53.61	45.82	46.37
240	48.77	54.26	48.87	52.03	45.27	46.30
264	64.51	67.92	64.01	64.80	61.12	60.59
288	67.33	70.07	66.28	66.92	64.26	63.60
312	69.40	72.13	68.61	69.76	67.06	66.16
336	73.30	73.89	71.82	73.41	70.96	69.62
360	73.84	73.42	72.27	73.95	71.31	70.13

Table 3 Corrosion rate data for HCS and FVA in 0.25 M H_2SO_4 solution at 0% to 3% GSO concentration

HCS							
GSO Conc. (%) / Exposure Time (h)	0% GSO	0.5% GSO	1% GSO	1.5% GSO	2% GSO	2.5% GSO	3% GSO
24	22.76	13.11	15.59	11.65	12.49	11.27	9.84
48	13.52	7.57	10.31	6.40	7.49	6.82	7.31
72	8.57	4.82	6.34	4.31	4.24	3.99	4.85
96	6.84	3.63	4.91	3.53	3.66	3.29	3.89
120	5.63	2.89	3.81	3.19	2.89	2.56	3.02
144	7.41	2.30	3.03	3.27	2.24	2.06	2.50
168	6.92	1.92	2.54	2.81	1.85	1.74	2.20
192	6.15	1.64	2.22	2.40	1.64	1.57	1.97
216	14.07	1.46	1.95	2.14	1.51	1.46	1.80
240	9.54	1.32	1.79	1.94	1.33	1.32	1.61
264	13.00	1.17	1.52	1.73	1.24	1.14	1.44
288	11.86	0.99	1.28	1.56	1.17	0.98	1.35
312	13.28	0.88	1.30	1.40	1.03	0.93	1.22
336	15.27	0.80	1.19	1.28	0.91	0.83	1.08
360	14.23	0.71	1.11	1.16	0.86	0.73	0.99
FVA							
GSO Conc. (%) / Exposure Time (h)	0% GSO	0.5% GSO	1% GSO	1.5% GSO	2% GSO	2.5% GSO	3% GSO
24	125.22	113.56	68.91	70.13	70.93	69.08	98.97
48	66.38	67.18	54.08	53.65	40.77	58.89	55.17
72	46.34	45.83	39.12	40.70	31.25	44.68	38.59
96	34.85	34.91	29.68	32.04	26.96	34.05	29.87
120	27.98	27.75	24.00	25.86	22.41	27.44	24.60
144	29.15	22.84	20.05	22.03	19.09	23.12	21.68
168	26.61	19.59	17.22	18.99	16.75	20.07	19.51
192	23.38	16.86	15.09	16.69	14.71	17.50	17.17
216	28.99	15.03	13.43	14.94	13.45	15.71	15.55
240	26.46	13.55	12.10	13.53	12.69	14.48	14.21
264	34.46	12.23	11.06	12.40	12.13	13.40	13.58
288	33.96	11.09	10.17	11.45	11.24	12.14	12.36
312	33.79	10.34	9.42	10.61	10.22	11.13	11.44

336	35.38	9.45	9.24	9.97	9.41	10.27	10.75
360	33.60	8.79	8.93	9.32	8.75	9.64	10.04

3.2 Statistical analysis

ANOVA statistical method was used to analyse the statistical importance and influence of GSO inhibitor concentration and exposure time on the corrosion inhibition performance of GSO on HCS and FVA alloys during the exposure hours. Table 5 depict the ANOVA results for HCS and FVA. On the table, statistical significance factor represents the numerical impact of GSO inhibitor concentration and time of exposure on the resistance of both alloys to corrosion. The theoretical significance factor represents a numerical factor wherewith the mean square ratio must be greater than, for the statistical significance factor to be relevant in the analysis. On Table 5, the mean square ratios for GSO concentration on HCS and FVA are significantly lower than the theoretical significance factor. Hence, this indicates GSO concentration is statistically irrelevant with regards to its impact on corrosion inhibition of the alloys. The corresponding statistical relevance factor for GSO concentration are 6.32%, and 2.38%%. The corresponding data for exposure time shows they are also statistically irrelevant with statistical relevance factor of -2197.74 and -291.11. These data show that while GSO inhibitor effectively inhibits HCS and FVA alloys in the acid solution with respect to concentration and exposure time, its performance is not statistically determinate.

Table 4 ANOVA data for GSO inhibition performance on HCs and FVA alloys in 0.25 M H_2SO_4 solution from 0% to 3% GSA concentration with respect to exposure time

HCS			
Source of Variation	**Mean Square Ratio (F)**	**Theoretical Significance Factor**	**Statistical Relevance Factor, F (%)**
GSO Concentration	0.02	2.16	6.32
Exposure Time	-4.80	2.03	-2197.74
FVA			
Source of Variation	**Mean Square Ratio (F)**	**Theoretical Significance Factor**	**Statistical Relevance Factor, F (%)**
GSO Concentration	0.06	2.16	2.38
Exposure Time	-3.74	2.03	-291.11

3.3 Standard deviation, mean and margin of error

Table 5 shows the standard deviation, mean inhibition value and margin of error for HCS and FVA inhibition efficiency data at all concentrations studied throughout the exposure hours. The mean inhibition values for HCS varied from 78.71% at 1.5% to 86.26% at 2.5% GSO concentration. Generally, the mean inhibition values for GSO on HCS varies around 80% inhibition efficiency signifying effective inhibition. The mean inhibition values for GSO on FVA varies from 49.62% at 2.5% to 56.73% at 1% GSO concentration. Generally, the mean inhibition values vary around 50% inhibition signifying poor inhibition performance. The SD values shows the deviations of GSO inhibition values from mean inhibition values for HCS and FVA alloys. GSO at 2% and 2.5% concentration exhibited the lowest SD values for HCS compared to other concentrations signifying thermodynamic stability at such concentrations. Nevertheless, the SD values for HCS where generally and significantly lower than the values obtained for FVA signifying greater unstable inhibition behavior of GSO on FVA compared to HCS. The total GSO inhibition efficiency data above 70% inhibition efficiency on HCS is 2% compared to 22% for FVA.

Table 5 Data for standard deviation, mean and margin of error for 304ST and F20ST alloys in H_2SO_4 solution at specific NaCl concentration

HCS						
Conc. (%)	**0.5%**	**1%**	**1.5%**	**2%**	**2.5%**	**3%**
SD	10.12	13.19	14.26	9.42	8.95	10.91
Mean	85.61	80.59	78.71	85.25	86.26	82.71
Margin of Error	9.79%		**Total Data above 70% Inhibition Efficiency**			82%
FVA						
Conc. (%)	**0.5%**	**1%**	**1.5%**	**2%**	**2.5%**	**3%**
SD	20.57	17.29	19.16	15.67	20.20	18.44

Mean	52.13	56.73	52.20	56.31	49.62	50.17
Margin of Error	10%		**Total Data above 70% Inhibition Efficiency**			22%

Conclusion

Grapeseed oil effectively inhibited corrosion of high carbon steel compared to ferrovanadium alloy. Inhibition efficiency data significantly varied and progressively increased with respect to exposure compared to differences in inhibitor concentration. Data from standard deviation showed inhibition performance of grapeseed oil on high carbon steel depicts greater thermodynamically and minimal variation from mean values compared to contrasting values from ferrovanadium alloy.

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