Protection performance of Azadirachta indica leaf essential oil on corrosion of plain carbon steel in weak HCl and NH_4Cl

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Abstract. *Azadirachta indica* leaf essential oil (ADI) was assessed for its corrosion protection properties on plain carbon steel within 0.5 M HCl, 3.74 M NH_4Cl, 1 M HCl, and 5.61 M NH_4Cl. Weight loss data indicates ADI exhibited strong inhibition properties in HCl, with peak performance of 97.53% and 97.55% at 5% ADI. The results indicates strong molecular interaction between protonated ADI molecules and the charged steel surface. ADI showed poor inhibition at 1-4% ADI within NH_4Cl, at values below 50%. At 5% ADI concentration, the data considerably rose to 78.52% and 78.0% after 312 h. This shows ADI performance in NH_4C is significantly concentration dependent which limits it versatility compared to its performance in HCl solution. Standard error for ADI performance within 0.5 M HCl were extreme, suggesting thermodynamic stability of inhibitor molecules. Standard error were lower in 1 M HCl, 3.74 M NH_4Cl, and 5.61 M NH_4Cl. Numerical data showed a portion of ADI protection performance values generally exceeded 70%. Statistical analysis showed immersion duration was more dominant on the performance of ADI compared to ADI concentration, while ADI concentration wasmore influential in NH_4Cl.

1 Introduction

Carbon steels are extensively utilized in petroleum refineries, fertilizer processing, mining of minerals, building technology etc. [1,2]. Their preference stems from being comparatively inexpensive, convenience of production, recoverability etc. Corrosion is frequently depicted as the chemical or electrolytic interplay of intermetallic compounds with their surroundings [3,4]. The deterioration mechanism is more common in settings with elevated anion concentrations, which react with intermetallic compounds. Their availability in industrial settings results in electrochemical degradation of the exterior characteristics of these iron alloy. Hydrogen chloride solution is among the top difficult common reagents to handle owing to its corrosive properties. The employment of carbon steels in these conditions negatively impacts their longevity and service life. Ammonium chloride is primarily utilized in industry as a nitrogen source for fertilizers. Other applications include the formulation of laboratory buffers and batteries. Ammonium chloride frequently causes corrosion in petrochemical equipment and piping systems. Employing chemical by-products referred to

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as corrosion inhibitors is the most effective and economical approach to managing corrosion [5]. Corrosion inhibitors function by inhibiting the electrochemical reactions that lead to corrosion [6,7]. Although most anti-corrosion agents are harmful, impractical and exorbitant, employing plant extracts as alternatives has yielded encouraging results in recent years [8-15]. This study focuses on analyzing the data related to the corrosion prevention property of *Azadirachta indica* leaf essential oil on plain carbon steel in a diluted solution of hydrochloric acid and ammonium chlorides.

2 Material and methods

A 12 mm diameter plain carbon steel (PCS) rod was partitioned into 6 representative parts with a typical length of 7 mm for the purpose of evaluating weight loss. Acid electrolyte solutions were prepared incorporating high-purity grade hydrochloric acid (HCl) and ammonium chloride (NH_4Cl) reagents into filtered H_2O, resulting in concentrations of 3.74 M NH_4Cl, 1 M HCl, 5.61 M NH_4Cl and 0.5 M HCl. *Azadirachta indica* leaf essential oil (ADI) delivered from NOW Foods USA was incorporated into the prepared solution at proportional content ranging from 1% to 6%. The PCS samples were placed in translucent containers containing the different electrolytes with varying concentrations of ADI. Weight measurements were conducted after every 24-hour break using the Ohaus system. The corrosion rate figures for PCS was estimated based on the computational formula provided in Equation (1);

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

W represents weight loss (g), D stands for density (g/cm^2), A signifies the area (cm^2), and T marks the measurement duration (h). The inhibition performance index (η) was estimated using a mathematical formula in Equation (2).

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

ω_1 demonstrates the weight reduction of PCS in the absence of extracts within the electrolyte, whereas ω_2 shows the weight reduction of PCS at specific extract content. A dual element monocomponent inferential statistical examination (F-test) was employed to determine the statistical significance of ADI percentage content and measurement duration on the extract performance index. The test was conducted at a reliability threshold of 95% (critical level of $\alpha = 0.05$) based on the computational formulas provided. The sum of squared values in columns (immersion duration) was estimated as shown below in Equation (3);

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

The sum of squared values in rows (ADI content) was estimated based on the computational formulas provided in Equation (4);

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

The cumulative squares total is given in Equation (5);

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

3 Results and discussion

3.1 Gravimetric analysis

The corrosion of plain carbon steel (PCS) in 3.74 M NH_4Cl, 0.5 M HCl, 5.61 M NH_4Cl and 1 M HCl at varying ADI concentrations (1%-5%) is detailed in Tables 1 and 2. Tables 3 and 4 provide the relevant data for protection performance figures of ADI in these acidic solutions

over different inspection periods. Initially, the corrosion rates of PCS inside 0.5 M HCl and 1 M HCl at 0% ADI concentrations were recorded at 0.0040 mm/y and 0.0145 mm/y (24 h), respectively. The corrosion rate in the 0.5 M HCl solution increased progressively, reaching 0.0086 mm/y at 312 h. The corrosion rate figures for PCS in 1 M HCl electrolyte are predominantly above those in 0.5 M HCl. The corrosion figures of PCS in 1 M HCl decreased to 0.0129 mm/y due to the weakening of the electrolyte. A marked decline in PCS corrosion was observed with 1% to 5% ADI content in 0.5 M HCl electrolyte. The final corrosion rate values for PCS ranged from 0.0002 mm/y to 0.0005 mm/y after 312 h of exposure. Similar trends were observed for PCS in 1 M HCl, with slightly higher values ranging from 0.0003 mm/y to 0.011 mm/y. The corrosion rate figures in 3.74 M NH_4Cl and 5.61 M NH_4Cl exhibited similar trends. However, the corrosion rate measurement for PCS at 0% ADI content were above those in 0.5 M HCl but lower than those in 1 M HCl solution. The corrosion rate measurement in 0.5 M HCl and 1 M HCl started at 0.00327 mm/y and 0.00439 mm/y (24 h) and decreased to 0.00048 mm/y and 0.00050 mm/y after 312 h.

Table 1. Corrosion rate information for PCS in 0.5 M and 1 M HCl solution in relation to immersion duration.

Acid Conc.	**0.5 M HCl**					**1 M HCl**				
Immersion	**ADI Conc. (%)**									
duration (h)	**1%**	**2%**	**3%**	**4%**	**5%**	**1%**	**2%**	**3%**	**4%**	**5%**
24	0.0029	0.0028	0.0027	0.0018	0.0010	0.0044	0.0034	0.0033	0.0032	0.0030
48	0.0015	0.0015	0.0012	0.0010	0.0006	0.0030	0.0017	0.0017	0.0016	0.0015
72	0.0011	0.0010	0.0009	0.0007	0.0004	0.0024	0.0012	0.0012	0.0011	0.0011
96	0.0009	0.0008	0.0007	0.0006	0.0003	0.0022	0.0010	0.0009	0.0009	0.0008
120	0.0009	0.0006	0.0006	0.0005	0.0003	0.0019	0.0008	0.0008	0.0007	0.0007
144	0.0008	0.0005	0.0005	0.0005	0.0003	0.0017	0.0007	0.0006	0.0006	0.0006
168	0.0007	0.0005	0.0005	0.0004	0.0003	0.0016	0.0006	0.0006	0.0006	0.0005
192	0.0007	0.0004	0.0004	0.0004	0.0003	0.0015	0.0006	0.0005	0.0005	0.0004
216	0.0006	0.0004	0.0004	0.0004	0.0002	0.0014	0.0005	0.0005	0.0005	0.0004
240	0.0006	0.0004	0.0004	0.0003	0.0002	0.0018	0.0005	0.0004	0.0004	0.0004
264	0.0006	0.0003	0.0003	0.0003	0.0002	0.0021	0.0004	0.0004	0.0004	0.0004
288	0.0005	0.0003	0.0003	0.0003	0.0002	0.0011	0.0004	0.0004	0.0004	0.0003
312	0.0005	0.0003	0.0003	0.0003	0.0002	0.0011	0.0004	0.0003	0.0003	0.0003

Table 2. Corrosion rate information for PCS in 3.74 M and 5.61 M NH_4Cl solution in relation to immersion duration.

Acid Conc.	**3.74 M NH_4Cl**					**5.61 M NH_4Cl**				
Immersion	**ADI Conc. (%)**									
Duration (h)	**1%**	**2%**	**3%**	**4%**	**5%**	**1%**	**2%**	**3%**	**4%**	**5%**
24	0.0029	0.0025	0.0018	0.0004	0.0000	0.0033	0.00265	0.0023	0.0020	0.0000
48	0.0015	0.0014	0.0010	0.0003	0.0000	0.0017	0.00136	0.0012	0.0011	0.0001
72	0.0011	0.0010	0.0007	0.0002	0.0000	0.0012	0.00093	0.0009	0.0008	0.0001
96	0.0008	0.0008	0.0006	0.0002	0.0001	0.0009	0.00072	0.0007	0.0006	0.0001
120	0.0007	0.0007	0.0005	0.0002	0.0001	0.0007	0.00060	0.0006	0.0005	0.0001
144	0.0006	0.0006	0.0004	0.0002	0.0001	0.0006	0.00054	0.0005	0.0004	0.0001
168	0.0005	0.0005	0.0004	0.0002	0.0001	0.0006	0.00048	0.0005	0.0004	0.0001
192	0.0005	0.0004	0.0004	0.0002	0.0001	0.0005	0.00044	0.0004	0.0004	0.0001
216	0.0004	0.0004	0.0003	0.0002	0.0001	0.0004	0.00040	0.0004	0.0004	0.0001
240	0.0004	0.0004	0.0003	0.0002	0.0001	0.0004	0.00038	0.0004	0.0004	0.0001
264	0.0004	0.0003	0.0003	0.0002	0.0001	0.0004	0.00037	0.0004	0.0004	0.0001
288	0.0004	0.0003	0.0003	0.0002	0.0001	0.0004	0.00035	0.0003	0.0003	0.0001
312	0.0004	0.0003	0.0003	0.0002	0.0001	0.0003	0.00033	0.0003	0.0003	0.0001

Table 3. Protection performance information for ADI on PCS in 0.5 M and 1 M HCl solution in relation to immersion duration.

Acid Conc.	0.5 M HCl					1 M HCl				
Immersion	ADI Conc. (%)									
Duration (h)	1%	2%	3%	4%	5%	1%	2%	3%	4%	5%
24	26.16	28.88	31.4	54.46	74.42	70.06	76.7	77.39	77.81	79.35
48	49.16	51.23	58.84	67.1	80	90.99	94.78	94.88	94.97	95.34
72	70.52	73.94	74.9	80.85	88.92	92.78	96.42	96.48	96.62	96.84
96	80.11	83.42	84.1	86.73	92.71	91.65	96.17	96.51	96.66	96.93
120	82.38	87.43	88.14	89.46	94.42	90.49	96.06	96.34	96.44	96.81
144	86.27	90.22	90.63	91.5	94.79	89.77	95.82	96.24	96.31	96.72
168	87.11	91.56	91.85	92.55	95.2	89.73	95.95	96.38	96.45	96.83
192	87.94	92.47	92.7	93.18	95.54	90.01	96.09	96.58	96.6	96.94
216	90.99	92.47	94.48	94.59	96.54	90.75	96.5	96.92	96.95	97.23
240	91.19	94.31	94.81	94.9	96.66	87.6	96.77	97.14	97.17	97.4
264	93.35	94.74	96.13	96.39	97.37	84.29	96.8	97.16	97.17	97.36
288	93.95	96.04	96.52	96.74	97.56	91.38	96.88	97.25	97.26	97.44
312	93.99	96.47	96.56	96.81	97.53	91.22	97.08	97.36	97.41	97.55

Table 4. Protection performance information for ADI on PCS in 3.74 M and 5.61 M NH_4Cl solution in relation to immersion duration.

Acid Conc.	3.74 M NH_4Cl					5.61 M NH_4Cl				
Immersion	ADI Conc. (%)									
Duration (h)	1%	2%	3%	4%	5%	1%	2%	3%	4%	5%
24	10.14	25.00	43.87	88.44	100.00	24.08	39.54	48.33	53.95	99.12
48	11.04	20.72	39.41	83.56	99.77	25.51	40.54	46.79	53.89	97.80
72	11.66	17.71	39.31	80.78	97.62	27.45	41.73	46.39	52.01	95.99
96	9.77	12.74	36.09	79.83	94.69	28.06	41.71	44.65	51.63	95.19
120	8.51	12.24	32.99	75.93	90.46	28.46	41.27	43.07	50.15	93.98
144	6.67	11.31	32.32	74.75	87.27	28.19	38.77	42.00	50.07	91.92
168	5.52	12.62	31.95	71.20	81.07	28.76	38.20	40.06	48.64	89.70
192	7.68	11.90	30.13	68.52	78.31	29.33	36.87	39.80	45.95	85.61
216	10.89	15.71	33.39	63.21	76.96	29.32	35.48	38.22	42.19	83.97
240	13.74	18.09	34.34	62.98	77.72	30.63	34.35	35.95	39.15	83.49
264	13.04	19.58	31.19	58.81	74.66	30.09	32.30	34.63	35.67	83.01
288	18.10	30.83	40.75	60.99	78.42	30.43	32.07	33.96	35.73	79.92
312	20.72	33.96	41.95	61.80	78.52	31.88	35.18	36.35	38.59	79.06

Amidst ADI content, the corrosion of PCS in NH4Cl decreased with increasing ADI content and immersion duration, resulting in corrosion rate measurement ranging from 0.00038 mm/y to 0.00010 mm/y in 3.74 M NH4Cl, and 0.00034 mm/y to 0.00011 mm/y in 5.61 M NH4Cl. Overall, the values indicate extract performance is dependent on % content and immersion duration. The performance index figures of ADI in 0.5 M HCl solution, as indicated in Table 3, were notably low from 1% to 4% after 24 h. However, at 5% ADI concentration, the value reached 74.42%. There was a gradual increase in protective performance values across all ADI concentrations, with final values generally exceeding 90%. In 1 M HCl solution, ADI demonstrated effective inhibition of PCS, with inhibition values surpassing 70% from the beginning of the exposure period. These values continued to rise over time, reaching levels generally above 90% inhibition. These findings suggest that ADI inhibition is more consistent in 1 M HCl solution compared to 0.5 M HCl solution. In 3.74 M NH_4Cl solution (Table 4), effective inhibition of PCS was observed at 4% and 5% ADI concentration (88% and 100%) after 24 h of exposure. In 5.61 M NH_4Cl solution, effective inhibition was achieved at 5% ADI concentration. The inhibition performance at

4% and 5% ADI concentration decreased to 61.80% and 78.5% after 312 h, respectively. The corresponding effective inhibition value for ADI in 5.61 M NH_4Cl solution (5% ADI concentration) decreased to 78.06%. Therefore, while ADI exhibited effective performance in 0.5 M and 1 M HCl electrolytes across all concentrations, effective inhibition was only observed at 4% and 5% ADI concentration in 3.74 M NH_4Cl solution, and at 5% ADI concentration in 5.61 M NH_4Cl solution.

3.2 Standard error, mean and error range

The standard error (SE), mean, and error range for ADI performance index on PCS in 0.5 M HCl, 3.74 M NH_4Cl, 5.61 M NH_4Cl and 1 M HCl electrolytes are presented in Tables 5 and 6. The mean performance index values from 0.5 M HCl and 1 M HCl (Table 5) indicate that ADI effectively mitigated the corrosion of PCS over time. The values from the 0.5 M HCl solution ranged from 79.5% to 92.44%, whereas the corresponding values from the 1 M HCl solution ranged from 88.5% to 95.6%. The SE from both HCl solutions illustrate the degree of fluctuation in performance index from the mean inhibition results. The SE data for ADI from the 0.5 M HCl electrolyte diminished with rising ADI content, while the corresponding values for ADI in the 1 M HCl solution were significantly lower from 5.9 at 1% ADI concentration to 4.9 at 5% ADI concentration. The corresponding values for ADI in the 1 M HCl solution are significantly lower. The margin of error indicates that 87.7% of ADI protection performance from the 0.5 M HCl solution have values greater than 70% protection index with error range of +7.99, while the protection performance values of ADI from the 1 M HCl solution exceeding 70% inhibition is 100% with error range of +0%. The average ADI protection performance values of 3.74 M and 5.61 M NH_4Cl in Table 6 are significantly lower tE results are comparatively low at all ADI concentrations from 3.74 M and 5.61 M NH_4Cl solution, indicating stability of ADI protection performance despite weak protection of PCS in the electrolytes. Table 6 shows that 30.8% of ADI protection performance data are above 70% inhibition effect in 3.74 M NH_4Cl compared to a corresponding value of 20% from 5.61 M NH_4Cl at error range of +11.22 and +9.72.

Table 5. Standard error, mean and error range for ADI protection performance data in 0.5 M and 1 M HCl electrolyte relative to exposure duration.

0.5 M HCl						**1 M HCl**					
ADI Conc. (%)	1%	2%	3%	4%	5%	**ADI Conc. (%)**	1%	2%	3%	4%	5%
SE	20.23	20.33	19.08	12.7	7.24	**SE**	5.9	5.5	5.4	5.3	4.9
Mean	79.47	82.55	83.93	87.3	92.4	**Mean**	88.5	94.8	95.1	95.2	95.6
Error Range	±7.99		**Data above 70% Performance**		87.68 %	**Error Range**	±0		**Data above 70% Performance**		100%

Table 6. Standard error, mean and error range for error for ADI protection performance data in 3.74 M and 5.61 M NH_4Cl electrolyte relative to exposure duration.

3.74 M NH_4Cl						**5.61 M NH_4Cl**					
ADI Conc. (%)	1%	2%	3%	4%	5%	**ADI Conc. (%)**	1%	2%	3%	4%	5%
SE	4.3	7.4	4.6	9.7	9.5	**SE**	2.1	3.4	4.8	6.9	7
Mean	11.3	18.7	36	71.6	85.8	**Mean**	28.6	37.5	40.8	46	89.1
Error Range	±11.22		**Data above 70% Performance**		30.8%	**Error Range**	±9.72		**Data above 70% Performance**		20%

3.3 Inferential statistical evaluation

Analysis of variance was utilized to assess the statistical significance of immersion duration and ADI concentration on the protective performance output of ADI extract. The findings of this analysis are presented in Table 7. The computational validity factor (CVF) indicates the percentage importance or effect of ADI concentration and immersion duration (independent variables) on the protective performance output of ADI. The mean squared estimate (MSE) represents the value that must exceed the conceptual impact factor (CIF) to established the validity of CVF.

Table 7. Inferential statistics for ADI protection performance out in 0.5 M HCl, 3.74 M NH_4Cl, 5.61 M NH_4Cl and 1 M HCl electrolyte.

0.5 M HCl				**3.74 M NH_4Cl**			
Cause of Deviation	**MSE (F)**	**CIF**	**CVF, *F* (%)**	**Cause of Deviation**	**MSE (F)**	**CIF**	**CVF, *F* (%)**
ADI Conc.	9.40	2.42	9.32	ADI Conc.	625.38	2.42	96.42
Immersion Duration	36.66	2.1	81.76	Immersion Duration	6.33	2.1	2.20
1 M HCl				**5.61 M NH_4Cl**			
Cause of Deviation	**MSE (F)**	**CIF**	**CVF, *F* (%)**	**Cause of Deviation**	**MSE (F)**	**CIF**	**CVF, *F* (%)**
ADI Conc.	121.47	2.42	15.80	ADI Conc.	642.72	2.42	96.95
Immersion Duration	283.60	2.1	83.02	Immersion Duration	5.00	2.1	1.70

The CVF with regards to ADI content in 0.5 M and 1 M HCl solutions are 9.32% and 15.80%, respectively, which, although relatively low, are statistically significant due to the corresponding MSE values surpassing the CIF. Conversely, the values for immersion duration in both HCl media are 81.76% and 83.02%, indicating that immersion duration is a predominant factor influencing the protective performance output of ADI extract. In NH_4Cl solution, the CIF for ADI concentration in 3.74 M and 5.61 M NH_4Cl solutions are 96.42% and 96.95%, while the corresponding values for immersion duration are 2.2% and 1.7%. All CVF values are deemed significant as the MSE values exceed the CIF. In conclusion, immersion duration exerts a substantial influence on the protective performance output of ADI on PCS in 0.5 M and 1 M HCl solutions, whereas ADI concentration predominantly affects the protective performance output of ADI in NH_4Cl solutions.

4 Conclusion

Azadirachta indica leaf essential oil demonstrated efficient corrosion protection properties on plain carbon steel corrosion in both HCl and NH_4Cl media, with the strongest inhibition occurring at the optimum inhibitor concentration in NH_4Cl media. Notably, in HCl media, the effectiveness of the oil in protecting against corrosion varied significantly over time, while in NH_4Cl media, the variation was more pronounced with different inhibitor concentrations. The standard deviation values were notably higher in 0.5 M HCl media, but lower and more consistent in NH4Cl, 5.61 M NH_4Cl, 1 M HCl and 3.74 M media. Statistical examination highlighted the impact of immersion duration and inhibitor concentration on the performance index of the *Azadirachta indica* leaf essential oil.

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