

Efficacy of Two Plant Extracts in inhibiting corrosion of mild steel under different media.**¹Ehujoo S. U., C.U.Ajuwa² and P.N.Atamuo²**

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Abstract

The study evaluated the efficacy of plant extracts as inhibitors of corrosion in mild steel using different environments, namely hydrochloric acid (HCl), tetraoxosulphate (VI) acid (H₂SO₄), atmosphere, distilled and sea water. Thirty six (36) mild steel coupons smeared with *Vernonia amygdalina* (bitter leaf) leaf extract and another set with *Azadirachta indica* (neem) leaf extract) were used for the study. Similar set of coupons unsmeared with either of the above extracts (control) was used for the investigation. Descriptive statistics were used in statistical analysis of collected data. Results showed that *Azadirachta* and *Vernonia* extracts recorded inhibitory efficiencies of 92.5% and 55.3%, respectively. Both plant extracts were more efficient in inhibiting corrosion of mild steel in H₂SO₄ medium. Corrosion rate on mild steel was a function of time, type of inhibitor and environment. Further studies should embark on other tropical plant extracts for purpose of comparison with known plant inhibitors.

Keywords: corrosion, metal, inhibition, plant extract.

Introduction

Mild steel which is widely used in industries are often exposed to the action of acid solutions. This is particularly seen in industrial processes involving picking, and acid cleaning. Consequently, the mild steel corrodes easily, requiring the use of inhibitors to slow down the decline in quality (Eddy *et al.*, 2008). Inhibitors create a barrier to corroding attack (El Ashry *et al.*, 2006). A good number of these inhibitors are toxic and expensive, calling for the need to explore alternative, environmental friendly and cheap inhibitors.

Popular alternatives are plant extracts as they are eco-friendly (Abiola *et al.*, 2007; Umoren, 2008) including use of some plant derivatives (Wang *et al.*, 2007). Plant extracts as inhibitors of corrosion are biodegradable and free from toxicants (Eddy and Odoemelam, 2009). Utilization of plant extract for inhibition of corrosion was reported in different environments (Oguzie, 2006). Neem (*Azadirachta indica*) and bitter leaf (*Vernonia amygdalina*) are plants found to possess phytochemicals such as saponins, tannins, flavonoids and anthraquinones. These phytochemicals are known to possess inhibitory properties. Nwabanne and Okafor (2001) found all the above phytochemicals in bitterleaf (*Vernonia amygdalina*). However, in a similar study, Eddy and Mamza, (2009)

reported that tannins, saponins, flavonoids and anthraquinones) are constituents in Neem (*Azadirachta indica*). Apart from the use of these plants in human and farm animal health, their use in suppressing corrosion was the focus of this study. However, the level of efficacy of the plants is necessary to enable end users to make a wise choice of one of the two options in minimizing corrosion of mild steel especially under different environments.

Based on the above, the major objective of the study was to estimate which one of the two plant extracts is most suitable for inhibiting corrosion of mild steel under different environment.

Materials and methods

Mild steel from universal steel company, Lagos with percentage composition of 0.0292 C, 0.0062 Si, 0.0152 S, 0.2529 Mn, 0.0096 P, 0.0261 Ni, 0.0125 Cr, 0.0869 Cu, 0.0047 Mo, 0.10060 As, 0.0101 Sn, 0.0030 Co, 0.0071 Al, 0.0005 Zn and 99.5350 Fe was used for the study. Solutions used were analytical grade reagent tetraoxosulphate (VI) acid (98% pure) to obtain 2.5 molar solution, 2.5 molar solution of hydrochloric acid, 2.5 molar solution of trioxonitrate (V) acid, distilled water, and sea water from the Atlantic ocean shore at Port Harcourt. Other materials used in the study were silicon carbide, grinding paper (grids of 60, 120, 220), fresh leaves of *Vernonia amygdalina* and *Azadirachta indica*. The study was conducted in confined room temperature.

Equipment used were electrons weighing scale balance, bench vice, plastic containers, measuring glass cylinders, funnels, small grinding machine, copper wires and sever net material.

Sample preparation

The mild steel was cut to form different coupons, each of dimension 50 x 40 x 11mm with 0.75mm hole and drilled for hanger and easy access of the coupons during removal (Fig. 1). Each coupon was decreased by washing with ethanol rinsed with acetone and allowed to dry in air. This was smeared with the inhibitor extracts of vernornia amygdalina and Azadriachta indica before it was allowed to dry in a dessicator at 40⁰C for prescrivation till it is used. Dried coupon were tagged with white cellophane tape.

The leaves of *Vernonia* and *Azadirachta* were obtained from the Federal University of Technology Owerri, Nigeria, where they were grinded and squeezed out on a bench vice from a sever net material before its use on the mild steel.

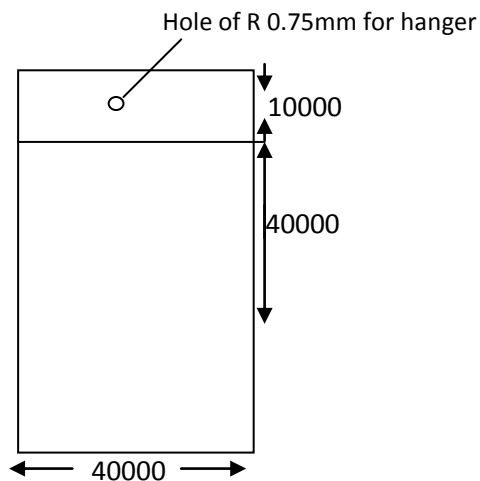


Fig. 1: Coupon dimension

Experiment

Thirty six (36) mild steel coupons smeared with *Vernonia amygdalina* were tagged BM¹ to BM³⁶. The same operation was done for *Azadirachta indica* to obtain DM₁ to DM₃₆. Then 36 mild steel coupons were not smeared but tagged NM₁ to NM₃₆ to represent control. These specimens were weighed on electron mass balance in the Department of Materials and Metallurgical Engineering Laboratory, Federal University of Technology, Owerri, Nigeria, and readings were recorded. Slots were cut on the cover of plastic containers (with a litre capacity) to accommodate 3 rows and six columns of coupons. The heights of containers were such that they accommodate the entire height of the coupon except the hanger which was above for easy access of coupons during removal. The experiment was arranged in 3 series viz;

Series A (Acid test on specimen), Series B (Test on sea and distilled water) and Series C (Exposure to the atmosphere).

In series A, the specimens were immersed in each of the acid solution used in the study, name

HCl, HNO₃ and H₂SO₄ at a concentration of 2.5M for each. Eighteen coupons were arranged in three rows and six columns. Two columns contained coupons smeared with a particular plant extract inhibitor while the last row was unsmeared. The acids were poured into the containers carrying the coupons and monitored at intervals of 4 hours for 24 hours.

In series B, the coupons were immersed in sea water and distilled water placed in separate transparent plastic containers. The same set-up as in Series A was put up, except for the timing and the duration of the test. The test was timed at 7 days interval for 42 days on mild steel coupons.

In series C, test was carried out by exposing the specimens to the atmosphere with the same duration as in Series B. The set-up was a little different. The coupons were tied to their holders and hung in an open place where only the atmospheric air came in contact with them. Care was taken not to allow rain water to come in contact with the coupons.



Fig 2: A fully sliced container

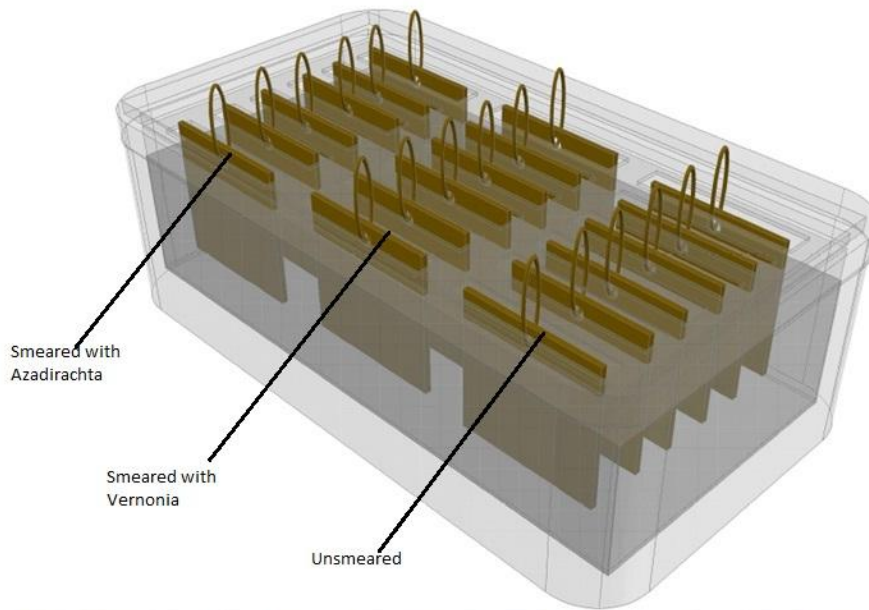


Fig.3: Container with coupons smearred with inhibitors, arranged and immersed in a medium

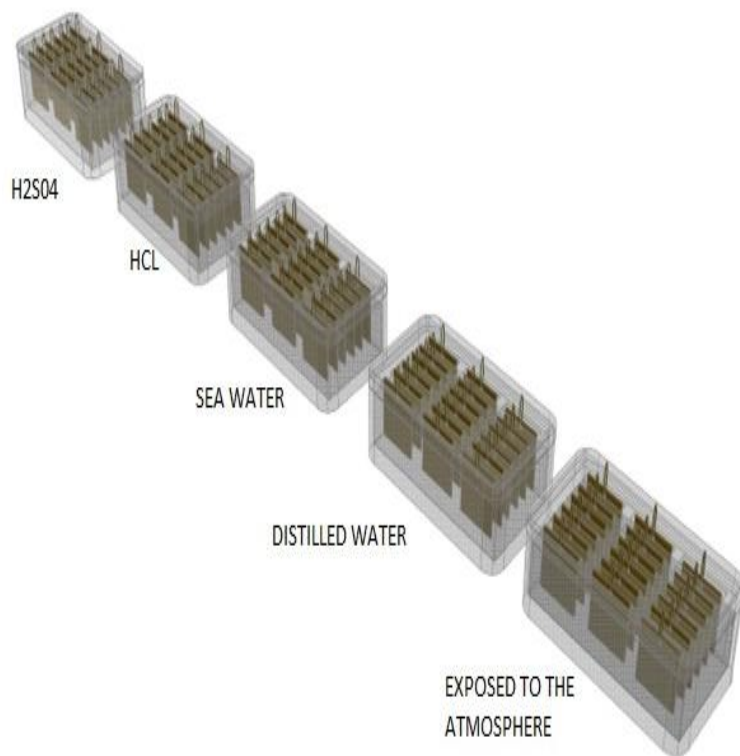


Fig.4: Arrangement of coupons in different media

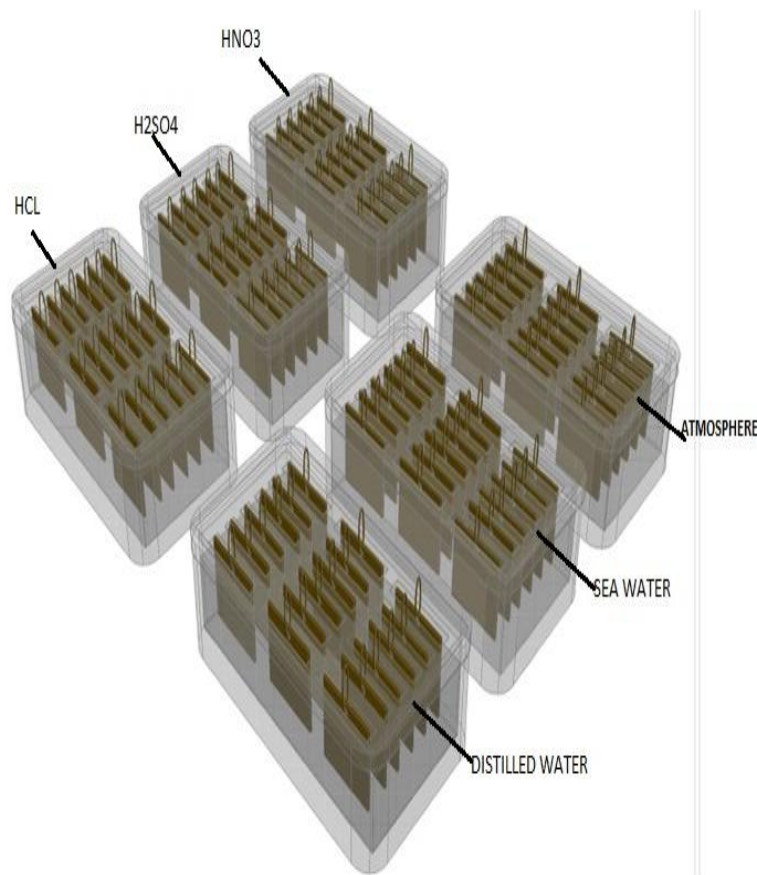


Fig.5: Final arrangement of coupons both smeared and un-smeared in different media

Measurements

Each sample was withdrawn from the test solutions, washed in a solution containing 50% sodium hydroxide and 100 grams per litre of zinc dust after every time interval (4 hours for series A, and 7 days for series B and C).

The washed samples were dried with acetone before re-weighing. The difference in weight for the interval was taken as the weight loss from the weight loss due to corrosion rate. Inhibition efficiency was thereafter calculated.

Calculations

Weight loss of the coupon (W_{loss}) was computed as follows:

$$W_{loss} = W_i - W_f$$

Where W_{loss} = weight loss(g)
 W_i = initial weight(g)
 W_f = final weight(g)

Corrosion rate of the acid on the coupon (C.R) was calculated as:

$$C.R = \frac{W}{A.T}$$

Where C.R = Corrosion rate
 W = Weight loss (mg)
 A = area (mm^2)
 T = time (hrs)

Efficiency of the inhibitors (E1) was computed as:

$$E1 = \left\{ \frac{C.R_{unsm} - C.R_{sm}}{C.R_{unsm}} \right\} \times 100/1$$

Where C.Rs = Corrosion rate of smeared (inhibitor)
 $C.R_{unsm}$ = Corrosion rate of unsmeared (control)

Results and Discussion

Weight loss of mild steel smeared with *Vernonia amygdalina* and *Azadirachta indica* on exposure for 24 hrs are shown on Table 1. Mean values of weight loss for the two plant extracts were 0.2098g (*Vernonia*), 0.448g (*Azadirachta*) and 0.1765g (control). However, weight loss ranged from 0.088 – 0.552 g (*Vernonia*), 0.081 – 1.182 g (*Azadirachta*) and 0.143 – 0.228 g (control). These values were under the medium of hydrochloric acid (2.5 mol). But, under the medium of tetraoxosulphate (VI) acid, mean values of weight loss were least in mild steel treated with plant extract as follows: 0.720g (*Vernonia*), 0.613 g (*Azadirachta*) and 1.082 g (Control) (Table 2). Values of weight loss ranged from 0.404 – 1.589 g (*Vernonia*), 0.031 – 1.509 g (*Azadirachta*) and 0.327 – 1.783 g (Control). The plant extracts were more effective in reducing corrosion of mild steel using tetra-oxo-sulphate (VI) medium.

Table 1. Variation of weight loss in grams(g) with exposure time(days) mild steel smeared with *Vernonia amygdalina* and *Azadirachta indica* juices immersed in acid

S/N	Test medium	Exposure time (Hours)	coupon Notation	Inhibitors	Initial weight (g)	Final weight (g)	weight loss (g)
1		4	BM1	<i>Vernonia</i>	13.662	13.574	0.088
2				<i>amygdalina</i>	13.168		
3		12	BM3		14.062	13.888	0.174
4		16	BM4		13.706	13.503	0.203
5		20	BM5		14.008	13.766	0.242
6		24	BM6		14.006	13.454	0.552
<i>Azadirachta indica</i>							
7	HCl (2.5mol)	4	DM1		14.101	14.02	0.081
8		8	DM2		14.113	13.959	0.154
9		12	DM3		13.99	13.824	0.166
10		16	DM4		14.463	13.984	0.479
11		20	DM5		13.969	13.362	0.607
12		24	DM6		14.939	13.757	1.182
Non Coated							
13		4	NM1	(Control)	14.071	13.928	0.143
14		8	NM2		14.277	14.126	0.151
15		12	NM3		14.506	14.339	0.167
16		16	NM4		14.189	14.01	0.179
17		20	NM5		13.883	13.692	0.191
18		24	NM6		13.897	13.669	0.228

Table 2. Variation of weight loss in grams(g) with exposure time(days) of mild steel smeared with *Vernonia amygdalina* and *Azadirachta indica* juices immersed in acid

S/N	Test medium	Exposure time (HOURS)	Coupon notation	Inhibitors	Weight loss (g)	Corrosion rate (mg/mm ² /yr)	Efficiency (%)
1	H ₂ SO ₄	4	BM7	Vernonia	0.314	204.662	3.976
2		8	BM8	Amygdalina	0.404	263.323	28.114
3		12	BM9		0.559	364.351	30.299
4		16	BM10		0.622	405.413	55.284
5		20	BM11		0.743	484.280	54.333
6		24	BM12		1.589	1035.694	10.881
<i>Azadirachta Indica</i>							
7		4	DM7		0.031	20.205	90.520
8		8	DM8		0.355	231.385	36.833
9		12	DM9		0.394	256.805	50.873
10		16	DM10		0.626	408.021	54.996

11	20	DM11	0.763	497.316	53.104
12	24	DM12	1.509	983.551	15.367

Non

Coated

13	4	NM7	0.327	213.135	
14	8	NM8	0.562	366.306	
15	12	NM9	0.802	522.736	
16	16	NM10	1.391	906.640	
17	20	NM11	1.627	1060.462	
18	24	NM12	1.783	1162.142	

Weight loss in mild steel treated with the plant extracts using seawater as the environment showed that the extracts especially from *Azadirachta indica* (mean= 0.486 g) was ineffective in inhibiting corrosion in 42 days (Table 3). Mean values were as follows: 0.282g (*Vernonia*), 0.486 g (*Azadirachta*) and 0.210 g (control) (Table 3). In Table 3, weight loss of mild steel ranged from 0.155 – 0.453 g (*Vernonia*), 0.154 – 1.504 g (*Azadirachta*) and 0.09 – 384 g (Control).

Table 4 shows results of weight loss in mild steel smeared with both plant extracts for 42 days in distilled water. Mean values of weight losses using

plant extracts were 0.362 g (*Vernonia*), 0.409 g (*Azadirachta*) and 0.172 g (control) under distilled water environment. Values of weight loss in mild steel ranged from 0.112 – 0.848 g (*Vernonia*), 0.152 – 0.867 g (*Azadirachta*) and 0.052 – 0.315 g (control).

Weight loss in mild steel treated with plant extracts for 42 days kept exposed to atmosphere varied with mean values of 0.376 g (*Vernonia*), 0.152 g (*Azadirachta*) and 0.223 g (Control) (Table 5). Ranges of weight in mild steel were 0.102 – 1.268 g (*Vernonia*), 0.117 – 0.186 g (*Azadirachta*) and 0.016 – 1.115 g (Control).

Table 3. Variation of weight loss in grams (g) with exposure time (days) of mild steel smeared with *Vernonia amygdalina* and *Azadirachta indica* juices immersed in sea water.

S/N	Test medium	Dates of removal	Exposure time (DAYS)	Coupon notation	Inhibitors	Initial weight (g)	Final weight (g)	Weight loss (g)
1	SEAWATER	9/7/2011	7	BM25	<i>Vernonia</i>	13.952	13.797	0.155
2		16/7/2011	14	BM26	<i>amygdalina</i>	13.937	13.703	0.234
3		23/7/2011	21	BM27		14.274	14.037	0.237
4		30/7/2011	28	BM28		14.346	14.07	0.276
5		6/8/2011	35	BM29		14.483	14.144	0.339
6		13/8/2011	42	BM30		13.926	13.473	0.453
					<i>Azadirachta indica</i>			
7		9/7/2011	7	DM25		14.234	14.08	0.154
8		16/7/2011	14	DM26		13.646	13.427	0.219
9		23/7/2011	21	DM27		13.795	13.527	0.268
10		30/7/2011	28	DM28		14.521	14.198	0.323
11		6/8/2011	35	DM29		14.146	13.698	0.448
12		13/8/2011	42	DM30		14.553	13.049	1.504
					Non Coated			
13		9/7/2011	7	NM25	(Control)	13.794	13.704	0.09
14		16/7/2011	14	NM26		14.006	13.863	0.143

15	23/7/2011	21	NM27	13.593	13.431	0.162
16	30/7/2011	28	NM28	13.7	13.487	0.213
17	6/8/2011	35	NM29	14.379	14.109	0.27
18	13/8/2011	42	NM30	14.276	13.892	0.384

Table 4. Variation of weight loss in grams (g) with exposure time (days) of mild steel smeared with *Vernonia amygdalina* and *Azadirachta indica* juices immersed in distilled water.

S/n	Test medium	Dates of removal	Exposure time (DAYS)	Coupon notation	Inhibitors	Initial weight (g)	Final weight (g)	Weight loss (g)
1	DISTILLED WATER	9/7/2011	7	BM19	<i>Vernonia</i>	14.512	14.4	0.112
2		16/7/2011	14	BM20	<i>Amygdalina</i>	14.854	14.704	0.15
3		23/7/2011	21	BM21		14.34	14.049	0.291
4		30/7/2011	28	BM22		13.626	13.322	0.304
5		6/8/2011	35	BM23		14.226	13.378	0.848
6		13/8/2011	42	BM24		14.115	13.65	0.465
					<i>Azadirachta</i>			
					<i>Indica</i>			
7		9/7/2011	7	DM19		14.565	14.413	0.152
8		16/7/2011	14	DM20		13.832	13.64	0.192
9		23/7/2011	21	DM21		14.675	14.366	0.309
10		30/7/2011	28	DM22		13.995	13.589	0.406
11		6/8/2011	35	DM23		13.959	13.432	0.527
12		13/8/2011	42	DM24		14.934	14.067	0.867
					Non Coated			
13		9/7/2011	7	NM19	(Control)	14.091	14.039	0.052
14		16/7/2011	14	NM20		13.831	13.748	0.083
15		23/7/2011	21	NM21		13.345	13.246	0.099
16		30/7/2011	28	NM22		13.756	13.558	0.198
17		6/8/2011	35	NM23		14.365	14.082	0.283
18		13/8/2011	42	NM24		14.445	14.13	0.315

Table 5. variation of weight loss in grams(g) with exposure time(days) of mild steel smeared with *Vernonia amygdalina* and *Azadirachta indica* juices exposed to the atmosphere

S/N	Test medium	Dates of removal	Exposure time (Days)	Coupon notation	Inhibitors	Weight loss (g)	Corrosion rate (mg/mm ² /yr)	Efficiency (%)
1	ATMOSPHERE	9/7/2011	7	BM31	<i>Vernonia</i>	0.102	1.271	-537.500
2		16/7/2011	14	BM32	<i>Amygdalina</i>	0.138	1.719	-375.862
3		23/7/2011	21	BM33		0.185	2.305	-374.359
4		30/7/2011	28	BM34		0.243	3.028	-395.918
5		6/8/2011	35	BM35		0.321	4.000	-248.913
6		13/8/2011	42	BM36		1.268	15.799	-13.722

Azadirachta

				Indica			
7	9/7/2011	7	DM31		0.117	1.458	-631.250
8	16/7/2011	14	DM32		0.124	1.545	-327.586
9	23/7/2011	21	DM33		0.147	1.832	-276.923
10	30/7/2011	28	DM34		0.164	2.043	-234.694
11	6/8/2011	35	DM35		0.174	2.168	-89.130
12	13/8/2011	42	DM36		0.186	2.318	83.318
				Non			
				Coated			
13	9/7/2011	7	NM31	(Control)	0.016	0.199	
14	16/7/2011	14	NM32		0.029	0.361	
15	23/7/2011	21	NM33		0.039	0.486	
16	30/7/2011	28	NM34		0.049	0.611	
17	6/8/2011	35	NM35		0.092	1.146	
18	13/8/201	42	NM36		1.115	13.893	

Table 5. variation of weight loss in grams(g) with exposure time(days) of mild steel smeared with *Vernonia amygdalina* and *Azadirachta indica* juices exposed to the atmosphere

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				Azadirachta				
				Indica				
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8		16/7/2011	14	DM32		0.124	1.545	-327.586
9		23/7/2011	21	DM33		0.147	1.832	-276.923
10		30/7/2011	28	DM34		0.164	2.043	-234.694
11		6/8/2011	35	DM35		0.174	2.168	-89.130
12		13/8/2011	42	DM36		0.186	2.318	83.318
				Non				
				Coated				
13		9/7/2011	7	NM31	(Control)	0.016	0.199	
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15		23/7/2011	21	NM33		0.039	0.486	
16		30/7/2011	28	NM34		0.049	0.611	
17		6/8/2011	35	NM35		0.092	1.146	
18		13/8/201	42	NM36		1.115	13.893	

Figure 6 indicates weight loss with exposure time of coupon in HCl smeared with plant extracts from *Vernonia* and *Azadirachta* together with the control (unsmear coupon). There was progressive loss in weight in the three different coupons irrespective of treatment.

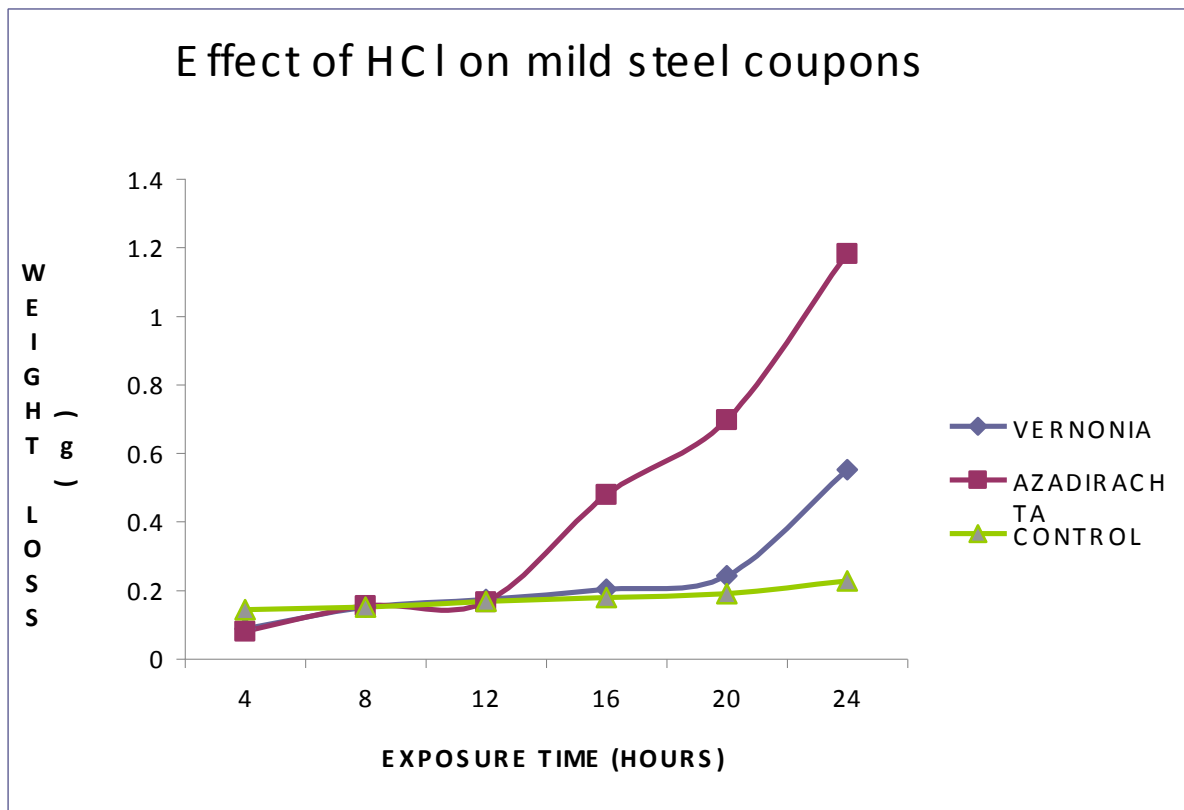


Fig. 6: Weight loss versus exposure time of coupons in HCl

The weight loss recorded in *Azadirachta indica* after 12 hours was rapid followed by *Vernonia* after 24 hours while weight loss in mild steel remained low in the control. This can be attributed to high concentration of chloride ion in the environment similar to the study earlier conducted by Phanasgaonkar *et al.* (1996).

There was more weight loss of mild steel from the un-smear coupon (control) and those of smear coupons under HCl (Fig. 6). Similar pattern

was followed by the plant extracts under H_2SO_4 medium. An initial decrease in weight loss with *Azadirachta* occurred at the commencement of the experiment, indicating that absorption took place initially. However, it was observed that coupon smear with *Vernonia* had higher weight loss than that with *Azadirachta*. Corrosion rate of coupon with exposure time using H_2SO_4 environment is shown in Fig 9. Similar distribution was recorded in Fig. 8.

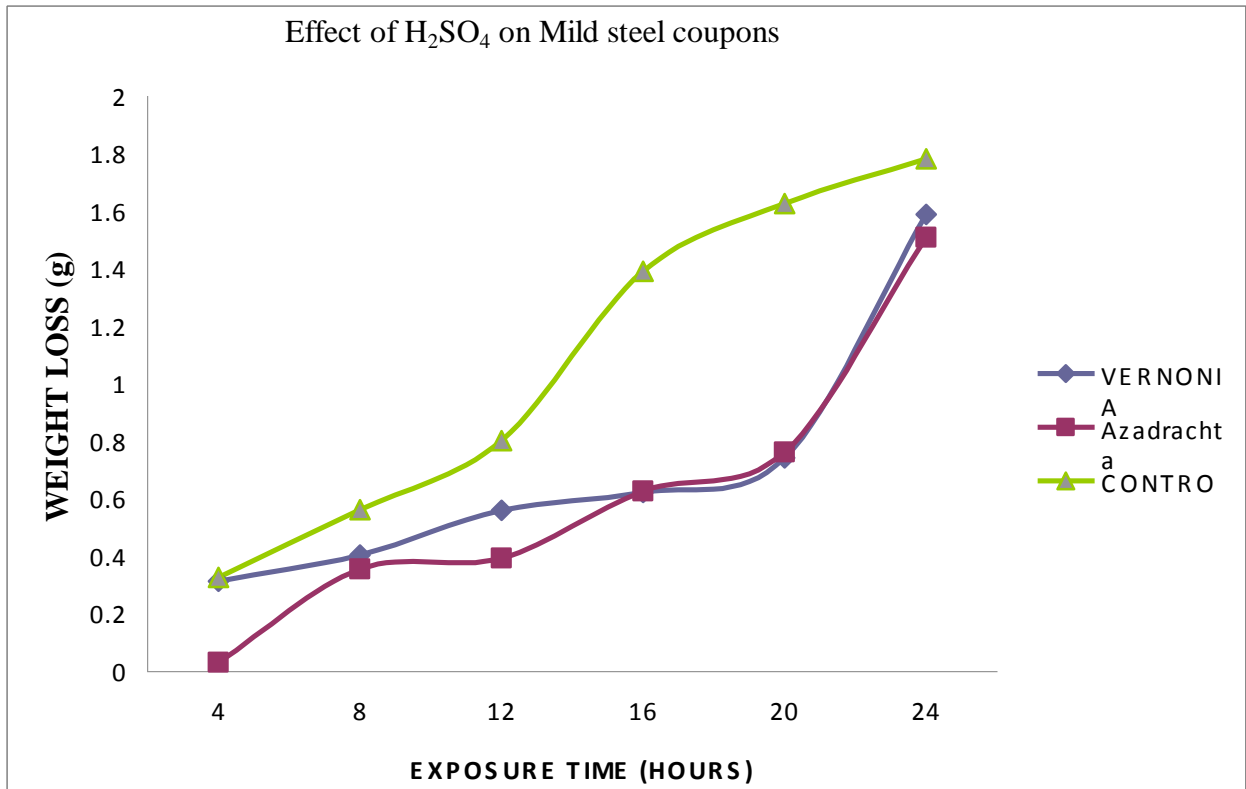


Fig 7: Weight loss versus exposure time in H₂SO₄

Initially, corrosion rate was low for *Azadirachta* followed by *Vernonia* than control with first 8 hours (Fig. 8). All the coupons had almost the same rate of corrosion at the 12th hour, but, the corrosion rate of *Azadirachta indica* rose rapidly

after 12 hours (Fig. 8). However, *Vernonia* rose above the control at 20th hour while the control maintained low rate of corrosion throughout the experiment. Low corrosion at the beginning of the experiment indicates absorption of plant extracts.

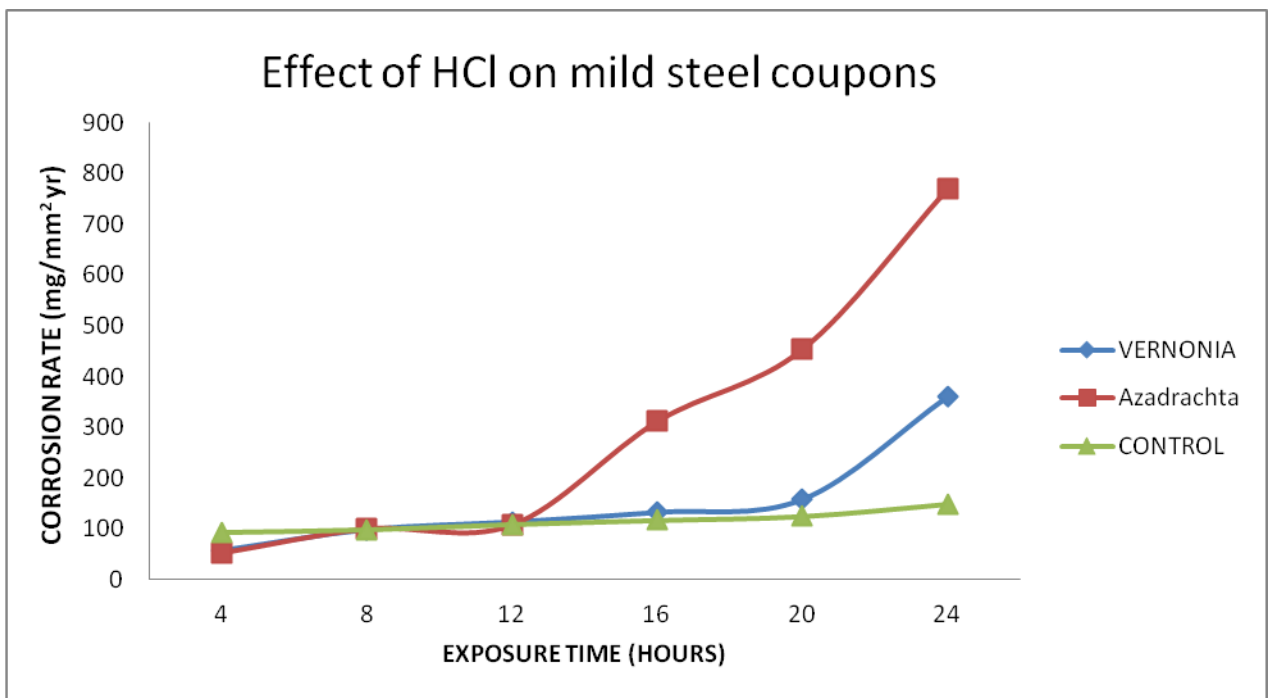


Fig 8: Corrosion Rate versus exposure time for Mild Steel in HCl

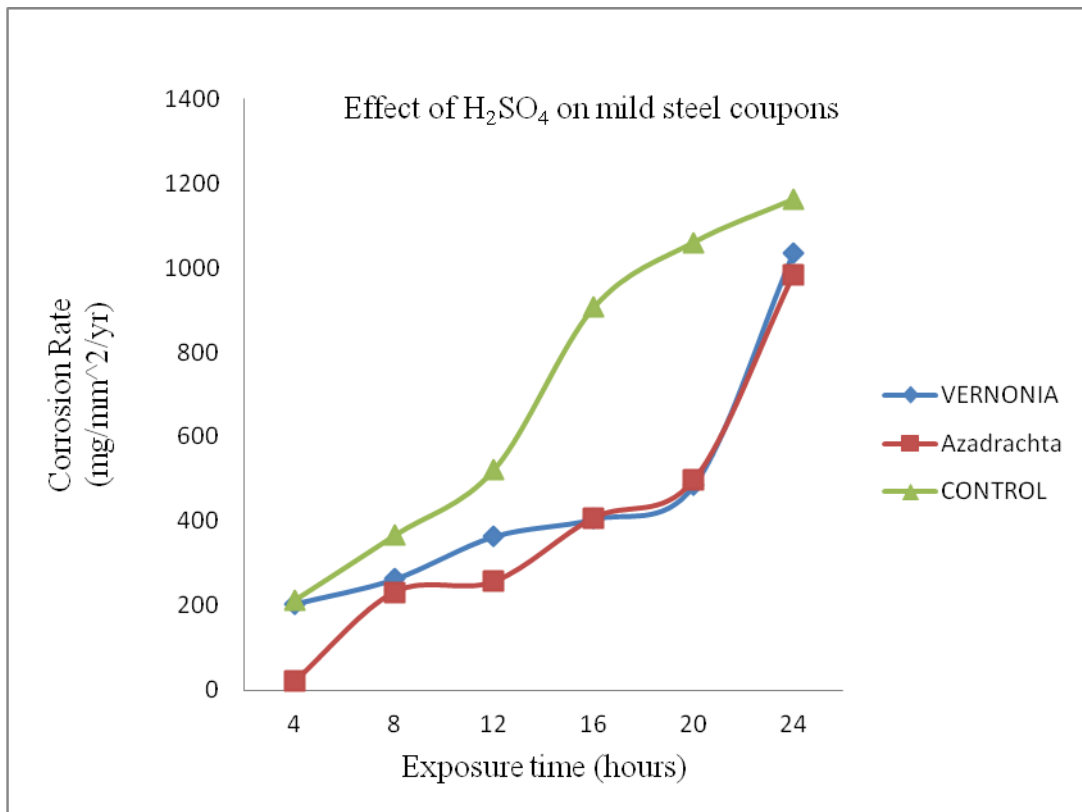


Fig 9: Corrosion Rate versus exposure time in H₂ SO₄

Inhibition efficiency is shown in Fig. 10 under the hydrochloric acid environment. The inhibition efficiency of *Azadirachta* was 43.36% while that of *Vernonia* stood at 38.46% at the first 4 hours. Inhibition efficiency of plant extracts

decreased substantially due to the chloride environment. It could be that the plant extracts were damaged by the protective oxide hence are not good inhibitors in a chloride environment.

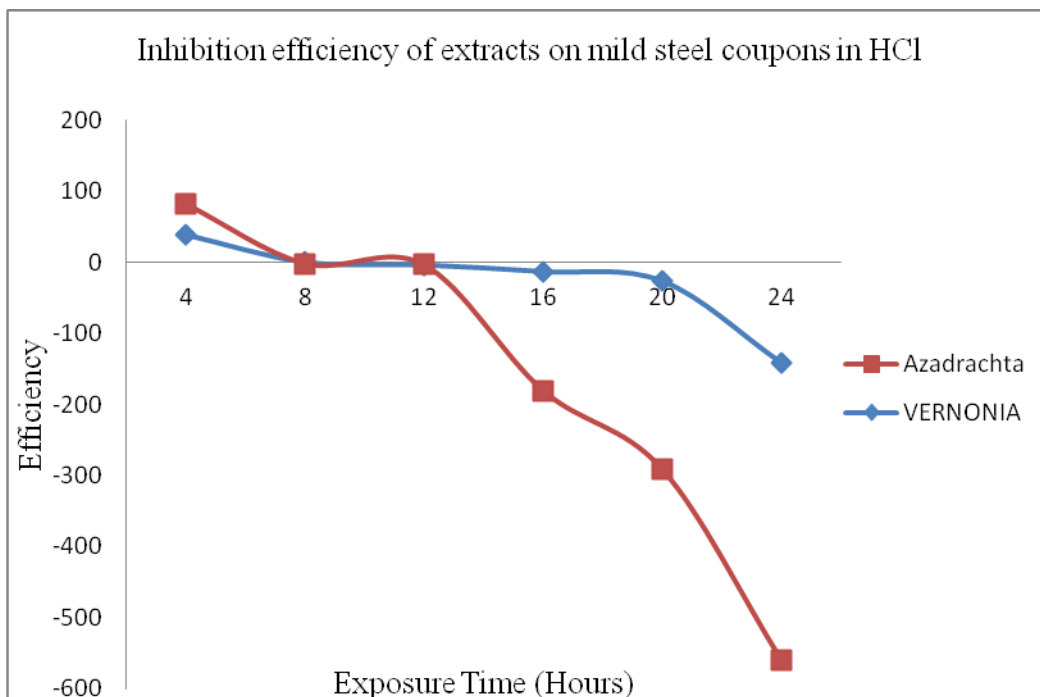


Fig 10: Efficiency of Inhibition versus exposure time in HCl

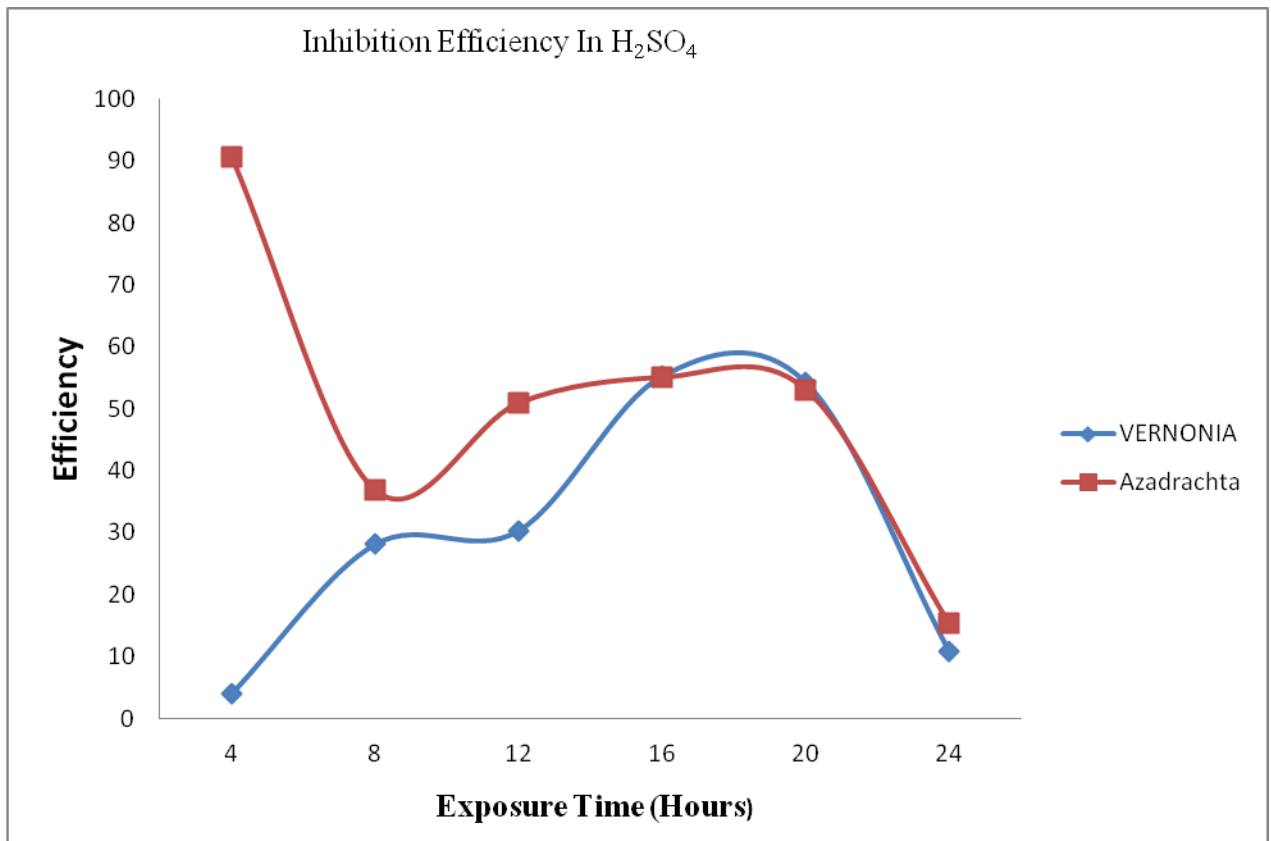


Fig 11: Shows inhibitive efficiency versus exposure time in H₂SO₄

Inhibitive efficiency of plant extracts under H₂SO₄ over 24 hours is given in Fig. 11. Although both inhibitors are effective in H₂SO₄ medium, *Azadirachta* (92.5%) was found to be more efficient than *Vernonia* (55.28%).

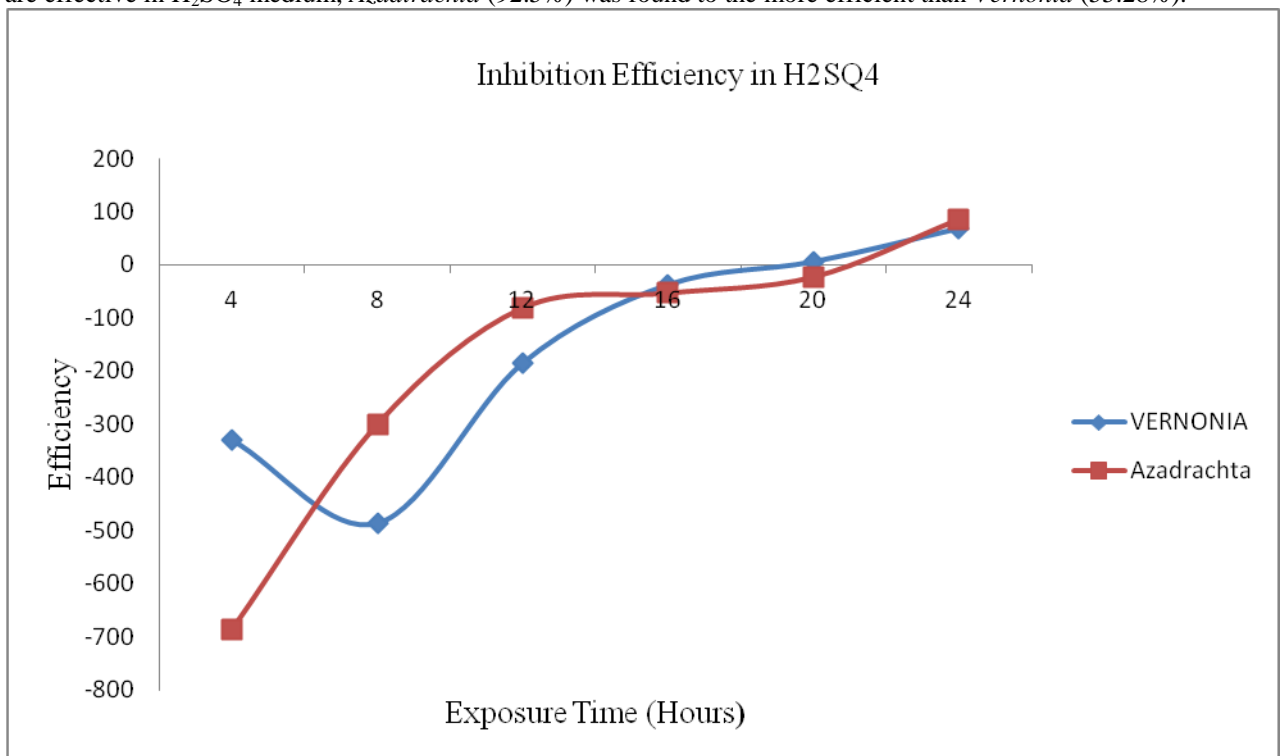


Fig. 11: Inhibitive Efficiency versus exposure time in H₂SO₄

The effectiveness of inhibitors depends on the relative concentration of chloride, inhibitor species and corrosion state of the steel at the time of treatment (Phanasgaonkar *et al.*, 1996). With the smearing of the plant extracts and drying, absorption has already taken place, and H₂SO₄ is believed to have strong affinity for water. Consequently, the inhibitors were able to inhibit corrosion of mild steel. In *Vernonia*, an increase in efficiency from a minimum of about 4% to 55.3% maximum after 20 hours is an indication that it reacted with the environment by electrochemical reduction to form product which is more effective. In *Azadirachta*, its decrease in efficiency is attributable to decrease in electrochemical reduction in line with findings of Thomas (1994).

Earlier studies showed that *Azadirachta* showed efficiency of 73.62% in H₂SO₄ (Eddy and Mamza, 2009) and 98.03% for *Vernonia* using alcohol extract (Odiogonyi, 2009). Given the result of inhibitory efficiency of 53.3% (*Vernonia*) and 92.5% (*Azadirachta*), one can deduce from this study that, both plant extracts can be used as inhibitors of corroding mild steel even without alcohol as the environment.

Conclusion

Plant extracts under H₂SO₄ medium exhibited greater inhibitory efficiency on corroding mild steel when compared other environments such as HCl and sea water. There was progressive loss in weight in mild steel coupons irrespective of treatment and medium. Corrosion rate differed with time among the plant extracts. Inhibition efficiency decreased substantially in HCl medium due to damage by chloride ions.

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