



# Experimental studies on *Areca catechu Spade* extract as corrosion inhibitor for Mild steel in acid medium

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**Abstract:** The influence of *Areca catechu Spade* (ACS) extract on the corrosion of mild steel in acid media was investigated by weight loss method, Tafel extrapolation, linear polarization and electrochemical impedance spectroscopy. The inhibition efficiency was increased with ACS extract concentration. SEM studies confirmed that the corrosion protection of mild steel surface was by the adsorption of inhibitors for ACS/HCl system.

**Key Words:** Corrosion, *Areca catechu Spade* (ACS), Mild steel (MS), Adsorption, Inhibitor,

## 1. INTRODUCTION:

Among the methods of corrosion control, use of inhibitors is very popular due to the ease of application. Mostly, the heterocyclic compounds containing O, S and N as heteroatoms serve as good inhibitors for corrosion [1]. However, most of the synthesized inhibitors are toxic to the environment. This has prompted the research for green corrosion inhibitors. Green corrosion inhibitors have a promising future for the quality of the environment because they do not contain heavy metals or other toxic compounds [2]. In addition, they are biodegradable and renewable source of materials. The objective of the present study is aimed at investigating inhibitive properties of acidic extracts of *Areca Catechu Spade* (ACS) for the corrosion of mild steel in 0.5M H<sub>2</sub>SO<sub>4</sub> and in 1M HCl.

## 2. MATERIALS:

### Specimen preparation

The mild steel used in the present work has the following composition



Mild steel specimens of rectangular size with area 1×5 cm<sup>2</sup> have been cut from a large sheet of mild steel and used for weight loss studies. Coupons of size 1 cm<sup>2</sup> were used for electrochemical studies and SEM analysis. The specimens were well cleaned and then degreased with acetone, prior to use.

### Selection of corrosive medium

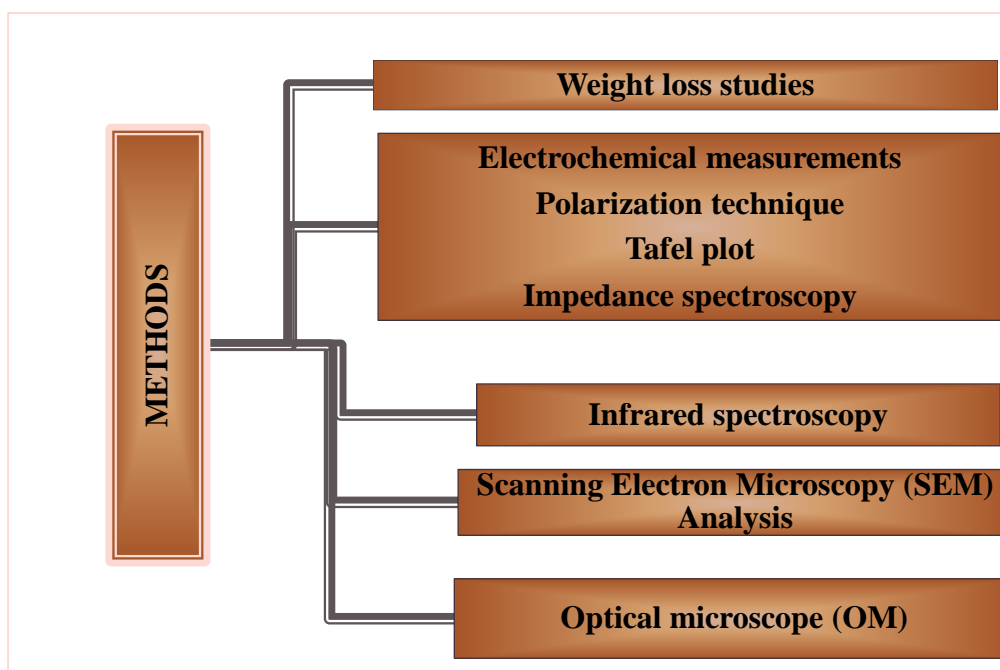
Acid pickling is an accepted treatment of a metallic surface to remove impurities, stains, steel rust, with an acid solution such as HCl and H<sub>2</sub>SO<sub>4</sub> [3], usually before subsequent processing such as extrusion, rolling, painting, galvanizing or plating with zinc, aluminium, etc. The present study is carried out in H<sub>2</sub>SO<sub>4</sub> and HCl medium.

### Preparation of inhibitor

In the present investigation, the eco -friendly inhibitor ACS was prepared by refluxing 25gms of ACS with 500ml of 0.5M H<sub>2</sub>SO<sub>4</sub> and also with 1M HCl for three hours and kept overnight for cooling purpose. The cooled extracts were filtered and made up to 500ml with 0.5M H<sub>2</sub>SO<sub>4</sub> and 1M HCl respectively to get 5% extract of inhibitors.

## 3. METHOD:

The following methods were adopted for the present study.



#### 4. RESULT AND DISCUSSION:

##### Weight loss method:

##### Effect of concentration and time of immersion for ACS

Dissolution of mild steel in 1M HCl and 0.5M H<sub>2</sub>SO<sub>4</sub> was studied for different concentrations (0.1% to 0.7%) of ACS extract at various period of immersion (½ h, 1h, 3h, 6h, 12h and 24h) at room temperature and the results are presented in Table 1 and Table 2 respectively. Results indicate as the concentration of ACS increases then inhibition efficiency also increases at all period of immersion [4]. Maximum inhibition efficiency was observed at 0.7% concentration of ACS extracts in H<sub>2</sub>SO<sub>4</sub> and HCl was around 93.6% and 94.3% respectively. Figure 1 and Figure 2 show the variation of IE with time of immersion in 0.5M H<sub>2</sub>SO<sub>4</sub> and 1M HCl respectively.

**Table 1. Effect of concentration and time on IE of MS/0.5 M H<sub>2</sub>SO<sub>4</sub> in presence of ACS**

S.No.	Conc (M)	1/2 h		1h		3h		6h		12h		24h	
		CR	IE (%)	CR	IE (%)	CR	IE (%)	CR	IE (%)	CR	IE (%)	CR	IE (%)
1	Blank	1247	-	1386	-	1812	-	1922	-	1468	-	1414	-
2	0.1	418	66.4	466	66.4	452	75.1	768	60.0	892	39.2	735	48.0
3	0.2	409	67.1	309	77.7	343	81.1	523	72.8	516	64.9	463	67.2
4	0.3	279	77.6	292	78.9	184	89.8	367	80.9	467	62.2	351	75.1
5	0.4	270	78.3	252	81.8	172	90.5	299	84.4	337	77.0	274	80.6
6	0.5	218	82.5	213	84.6	159	91.7	266	86.1	292	80.1	238	83.1
7	0.6	183	85.3	200	85.5	132	92.7	233	87.9	264	82.0	203	85.6
8	0.7	165	86.7	174	87.4	116	93.6	203	89.4	229	84.4	126	91.1

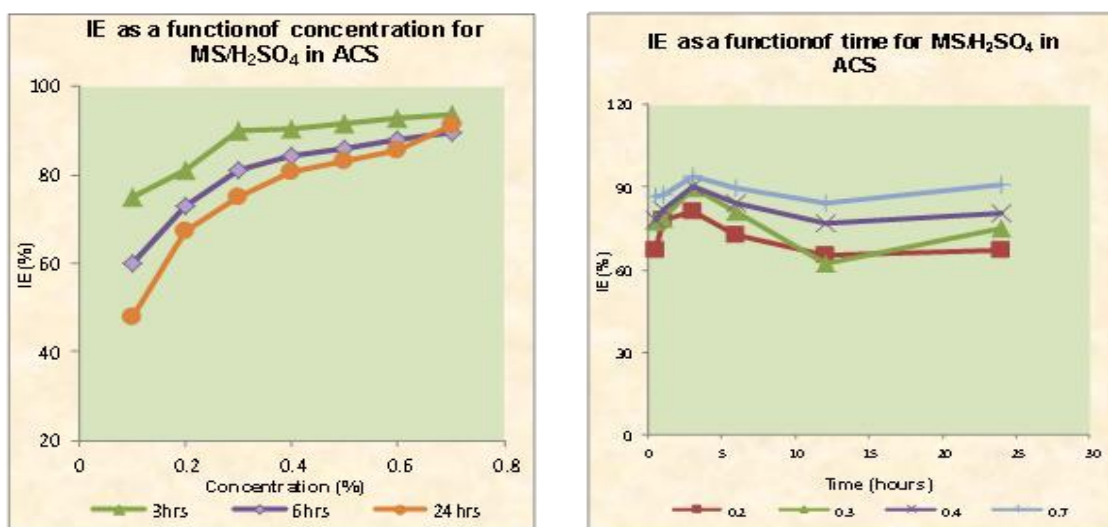


Figure 1 IE as a function of concentration and time for MS/H<sub>2</sub>SO<sub>4</sub> in ACS

Table 2 Effect of concentration and time on IE of MS/1M HCl in presence of ACS

S.No.	Con.c (M)	1/2 h		1h		3h		6h		12h		24h	
		CR	IE (%)	CR	IE (%)	CR	IE (%)	CR	IE (%)	CR	IE (%)	CR	IE (%)
1	Blank	1064	-	1434	-	3341	-	2395	-	2646	-	1465	-
2	0.1	488	54.1	423	70.5	1462	56.2	689	71.2	740	72.0	709	51.6
3	0.2	444	58.2	366	74.5	1045	68.7	457	80.9	520	80.3	508	65.3
4	0.3	427	59.8	305	78.7	768	77.0	349	85.4	283	89.3	449	69.3
5	0.4	418	60.7	261	81.8	661	80.2	266	88.9	252	90.4	398	72.8
6	0.5	401	62.3	252	82.37	594	82.2	216	91.0	201	92.4	353	75.9
7	0.6	392	63.1	226	84.2	517	84.5	204	91.5	152	94.2	302	79.3
8	0.7	366	65.6	204	85.7	514	84.6	162	93.2	151	94.3	289	80.2

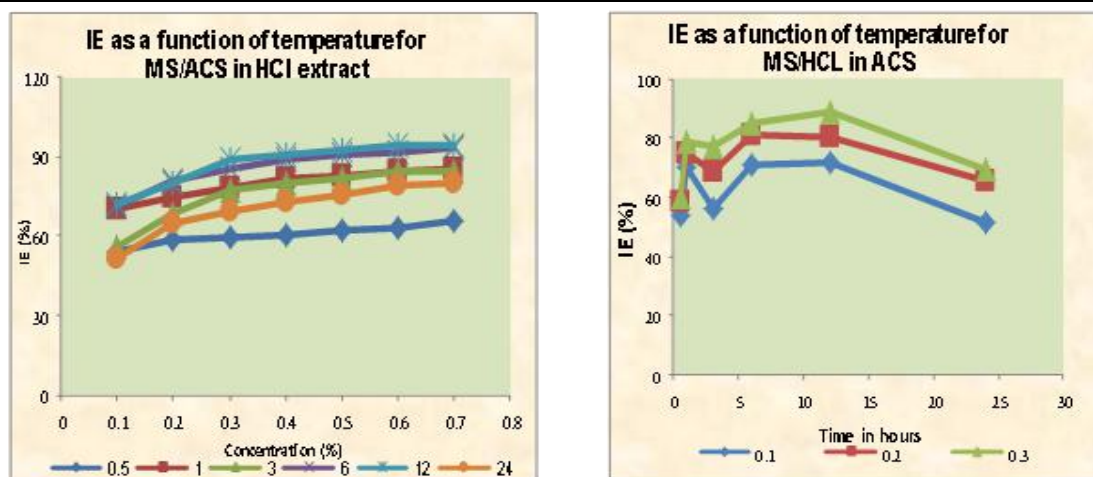


Figure 2 IE as a function of concentration and time for MS/HCl in ACS

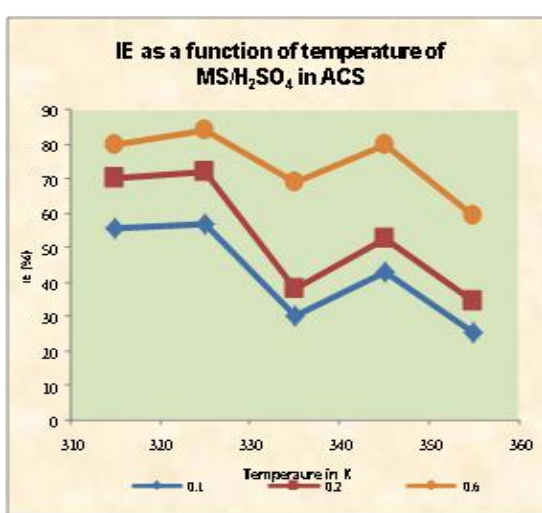
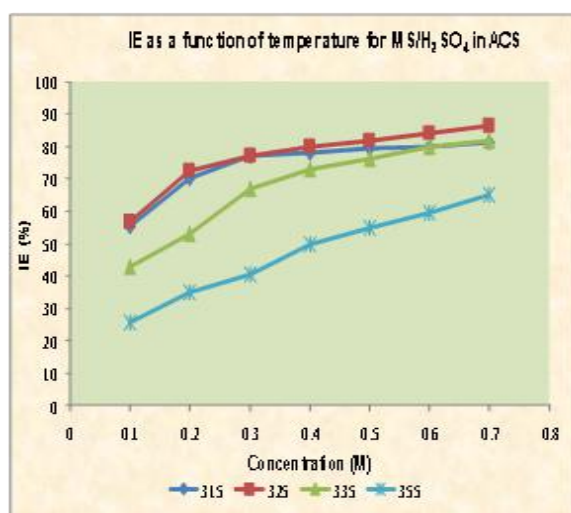


### Effect of temperature on mild steel corrosion in the presence of ACS extracts

Temperature can influence the interaction between MS surface and the acid medium in presence and absence of *Areca Catechu* Spade extract. The increase in IE with temperature from 315K to 325K, and 345K (81.2%, 86.6% and 81.8% respectively) (shown in Table 3 and Figure 3) indicates that adsorption of inhibitor ACS on MS surface was enhanced in H<sub>2</sub>SO<sub>4</sub> solution. Then at 335K and 355K the IE were found to decreased (75.9% and 65.0%), this may due to desorption of inhibitor molecules. The effect of temperature on the inhibited acid-metal reaction is very complex. As many changes occur on the metal surface such as rapid etching, desorption of inhibitor or the inhibitor may undergo decomposition at various higher temperatures [5]. As temperature increases from 315K to 355K, then the value of IE was changed for ACS inhibitor in MS/HCl bath (shown in Table 4 and Figure 4). That is IE increase from 72.8% (315K) to 89.0% (325K) and after that inhibition efficiency values were stabilized to 86.7% at 355K. Adsorption and desorption of inhibitor molecules continuously occurs at the metal surface and an equilibrium exist between these two processes at a particular temperature. With the increase of temperature the equilibrium between adsorption and desorption processes were shifted, these leading to a higher desorption rate than adsorption until equilibrium was again established at a different value of equilibrium constant.

**Table 3 Effect of temperature on the IE of MS/0.5 M H<sub>2</sub>SO<sub>4</sub> in presence of ACS**

S.no.	Con.c	315K		325K		335K		345K		355K	
		CR	IE (%)	CR	IE (%)	CR	IE (%)	CR	IE (%)	CR	IE (%)
1	Blank	6305	-	9934	-	10884	-	24953	-	36056	-
2	0.1	2808	55.4	4291	56.8	7596	30.2	14260	42.9	26898	25.4
3	0.2	1875	70.3	2756	72.3	6742	38.1	11765	52.9	23557	34.7
4	0.3	1430	77.3	2285	77.0	6558	39.7	8250	66.9	21429	40.6
5	0.4	1378	78.1	2006	79.8	5276	51.5	6776	72.8	18010	50.0
6	0.5	1282	79.7	1805	81.8	4378	59.8	5913	76.3	16318	54.7
7	0.6	1264	79.9	1596	83.9	3392	68.8	4971	80.1	14626	59.4
8	0.7	1186	81.2	1334	86.6	2625	75.9	4552	81.8	12611	65.0

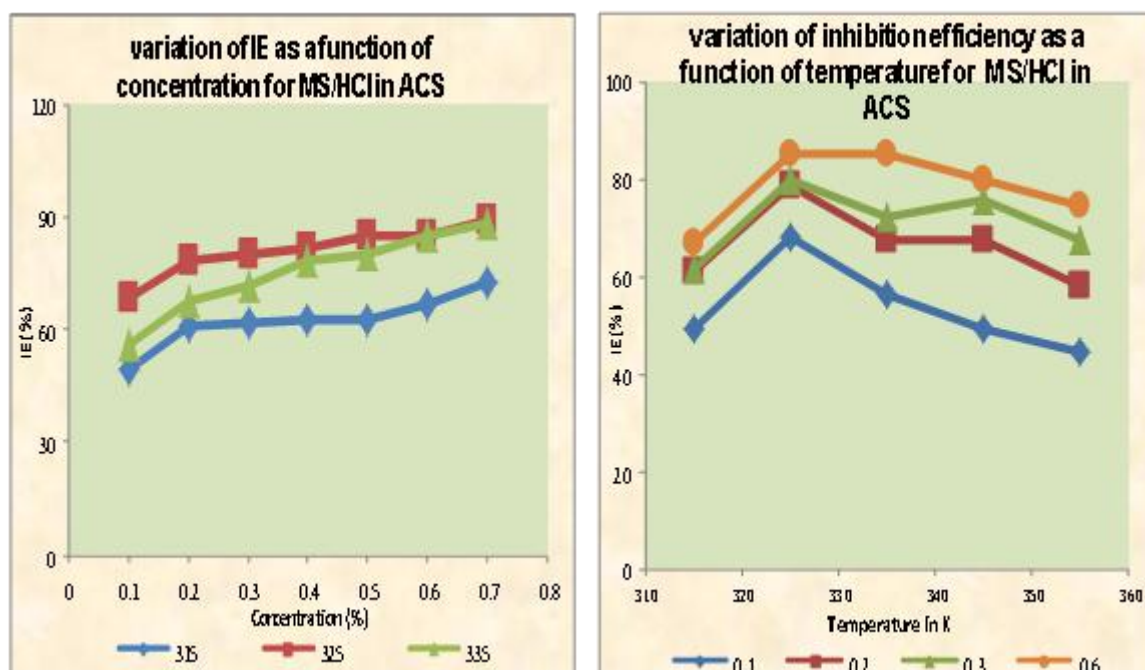


**Figure 3 IE as a function of concentration and temperature for MS/H<sub>2</sub>SO<sub>4</sub> in ACS**



**Table 4 Effect of temperature on the IE of MS/1MHCLin presence of ACS**

S.no.	Con.c	315K		325K		335K		345K		355K	
		CR	IE (%)	CR	IE (%)	CR	IE (%)	CR	IE (%)	CR	IE (%)
1	Blank	12123	-	11661	-	15333	-	23932	-	48458	-
2	0.1	61402	49.4	3724	68.1	6733	56.1	12132	49.3	26732	44.8
3	0.2	4709	61.1	2468	78.8	4954	67.7	7683	67.8	19912	58.0
4	0.3	4640	61.7	2363	79.7	4308	72.0	5765	75.9	15821	67.4
5	0.4	4544	62.5	2093	82.1	3349	78.2	5756	76.0	12760	73.7
6	0.5	4491	63.0	1779	84.7	3113	79.7	5573	76.7	12481	74.2
7	0.6	4003	67.0	1700	85.4	2302	85.0	4762	80.1	12193	74.8
8	0.7	3296	72.8	1255	89.2	1831	88.1	4430	81.5	6454	86.7



**Figure 4 IE as a function of concentration and temperature for MS/HCl in ACS**

### Electrochemical measurements

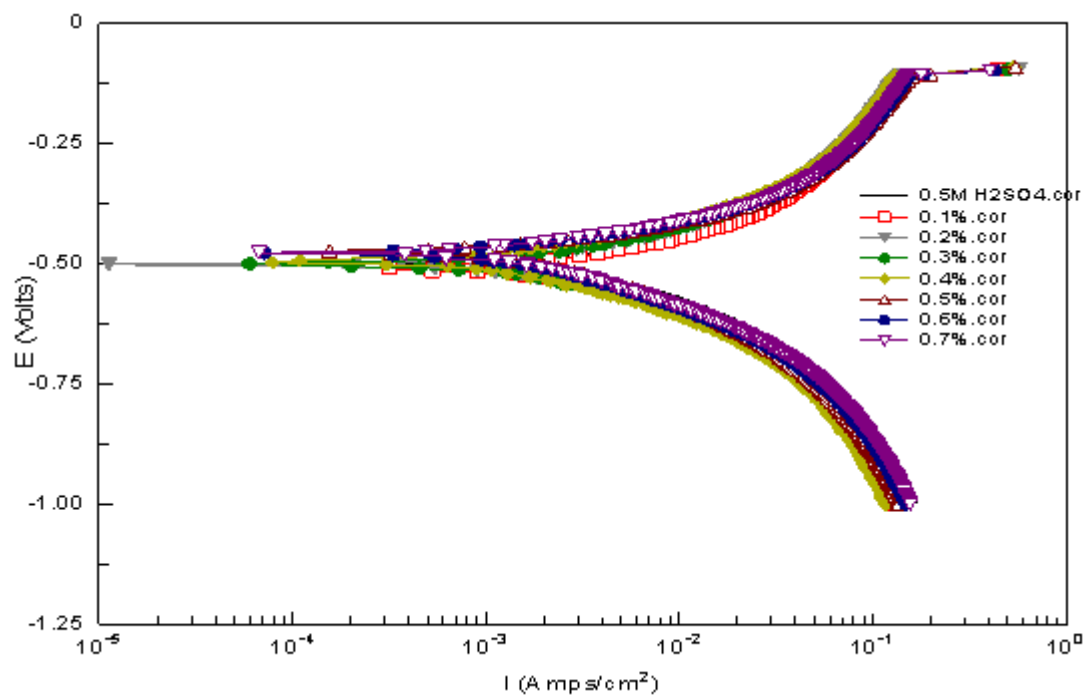
Polarization curves for mild steel at various concentrations of inhibitors ACS/ H<sub>2</sub>SO<sub>4</sub> and ACS/HCl in acidic solutions were shown in Figure 5 and 6. The extrapolation of Tafel straight line allows the corrosion current density ( $I_{corr}$ ). The values of ( $I_{corr}$ ), corrosion potential ( $E_{corr}$ ), cathodic and anodic Tafel lines ( $b_a$ ,  $b_c$ ) and inhibition efficiency were given in Table 5 and Table 6. From the Figure 5 and 6, it was clear that both the cathodic and anodic reactions are inhibited and IE increased as the inhibitor concentration increases in both acid media. It is clear that there was no definite trend in the shift of  $E_{corr}$  values, in the presence of various concentrations of ACS in 1M HCl and 0.5M H<sub>2</sub>SO<sub>4</sub> solution. The results implied that ACS can be classified as mixed type of inhibitor[6], predominantly cathodic in 1M HCl and



anodic in 0.5M H<sub>2</sub>SO<sub>4</sub> solution. Maximum IE using ACS in H<sub>2</sub>SO<sub>4</sub> medium was found to be 77.7% and in HCl medium IE was found to be 54.3%.

**Table 5 Tafel polarization measurement of MS/0.5 M H<sub>2</sub>SO<sub>4</sub> in presence of ACS**

S.no.	Concentration (%)	E <sub>corr</sub> (mv/sec)	I <sub>corr</sub> (Amp/cm <sup>2</sup> )	b <sub>a</sub> (mv/dec)	b <sub>c</sub> (mv/dec)	IE (%)
1	Blank	-0.49836	0.0050947	199	159	-
2	0.1	-0.50653	0.0034558	189	130	32.2
3	0.2	-0.49893	0.0032258	121	119	36.7
4	0.3	-0.47557	0.0027526	200	113	46.0
5	0.4	-0.49995	0.0016142	132	104	68.3
6	0.5	-0.49577	0.0014562	138	108	71.4
7	0.6	-0.47382	0.0010913	125	83	78.6
8	0.7	-0.47376	0.0011341	123	84	77.7



**Figure 5 Potentiodynamic polarization curves of MS/0.5M H<sub>2</sub>SO<sub>4</sub> in the presence of ACS**

**Table 6 Tafel polarization measurement of MS/HCl in the presence of ACS**

S.no.	Concentration (%)	E <sub>corr</sub> (mv/sec)	I <sub>corr</sub> (Amp/cm <sup>2</sup> )	b <sub>a</sub> (mv/dec)	b <sub>c</sub> (mv/dec)	IE (%)
1	Blank	-0.50229	0.0051106	141.35	123.95	-
2	0.1	-0.48598	0.0033449	129.9	157.62	34.6
3	0.2	-0.48711	0.0028267	121.9	148.22	44.7
4	0.3	-0.48849	0.0027856	119.26	145.64	45.5



5	0.4	-0.48953	0.0027791	124.1	138.1	45.5
6	0.5	-0.48775	0.0027175	122.73	145.11	46.8
7	0.6	-0.48462	0.0025029	116.9	132.71	51.0
8	0.7	-0.49049	0.0023348	110.81	105.49	54.3

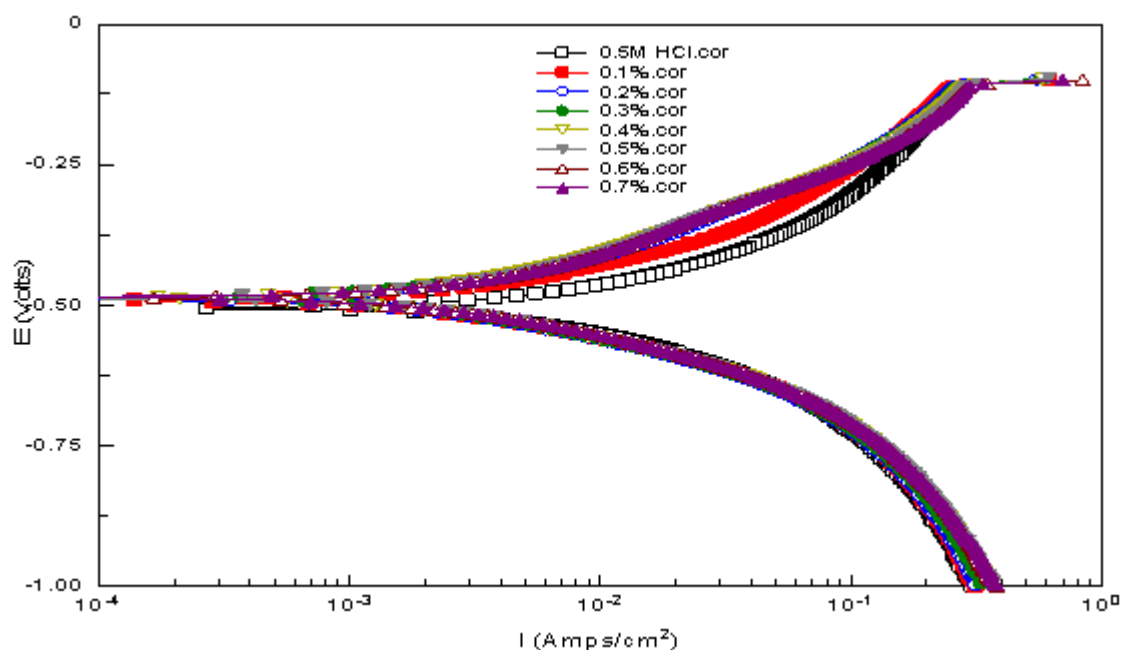


Figure 6 Potentiodynamic polarization curves of MS/1M HCl in ACS

### Linear polarization measurement

Linear polarization resistance ( $R_p$ ) values obtained from LPR method of the free acid solution and inhibitor acid solution containing highest concentrations of ACS. It was shown an increase in value from 7 to 12 in  $H_2SO_4$  solution and increase from 26 to 40 in HCl for ACS. (Table 7 & 8)

Where,  $R_{p(\text{inhibited})}$  and  $R_{p(\text{blank})}$  are linear polarization resistance in the presence and absence of the inhibitor respectively.

### Electrochemical Impedance measurements

Nyquist and Bode modulus plots for mild steel in 0.5 M  $H_2SO_4$  and 1 M HCl (with and without ACS) are presented in Figure 7a & 7b and Figure 8a & 8b respectively. The corresponding impedance parameters are given in Table 7 and Table 8. As it can be seen from tables, the  $R_{ct}$  (charge transfer resistance) values increased with increasing concentration of the inhibitor, which indicates that charge transfer process was the main cause for controlling mild steel corrosion. On the other hand, the values of  $C_{dl}$  (double layer capacitance) decrease with an increase in the inhibitor concentration [7], this was due to an increase in surface coverage by the inhibitors, which results higher value of inhibition efficiency. It is apparent from Nyquist plot that the impedance response of mild steel in HCl and  $H_2SO_4$  solution has significantly changed after the addition of ACS extracts in corrosive medium and the impedance of inhibited substrate increases with increasing concentration of inhibition in acid media.  $R_{ct}$  values for ACS extract at 0.7% concentration were observed that  $28.8 \Omega\text{cm}^2$  in  $H_2SO_4$  and  $64.3 \Omega\text{cm}^2$  in 1M HCl solution.

Table 7  $R_{ct}$ ,  $C_{dl}$ , and R for MS/  $H_2SO_4$  in presence of ACS

Sl.no	Conc(%)	$R_{ct}(\Omega\text{cm}^2)$	IE(%)	$R_p(\Omega/\text{cm}^2)$	IE(%)	$C_{dl}(f/\text{cm}^2)$	$\Theta$
1	Blank	6.9761	16.6	7.34		0.00028661	
2	0.1	8.3643	22.7	9.5021	22.8	0.00091798	-2.2029



3	0.2	11.5	39.3	6.9359	-6.69	0.00034676	-0.2097
4	0.3	25.416	72.6	13.918	57.5	0.00062496	-0.2099
5	0.4	26.1	73.3	17.274	37.0	0.00089192	-2.1120
6	0.5	26.917	74.1	11.645	46.0	0.00031022	-0.0824
7	0.6	27.021	74.2	12.252	40.1	0.00023581	0.1772
8	0.7	28.807	75.8	12.397	40.8	0.00011559	0.5967

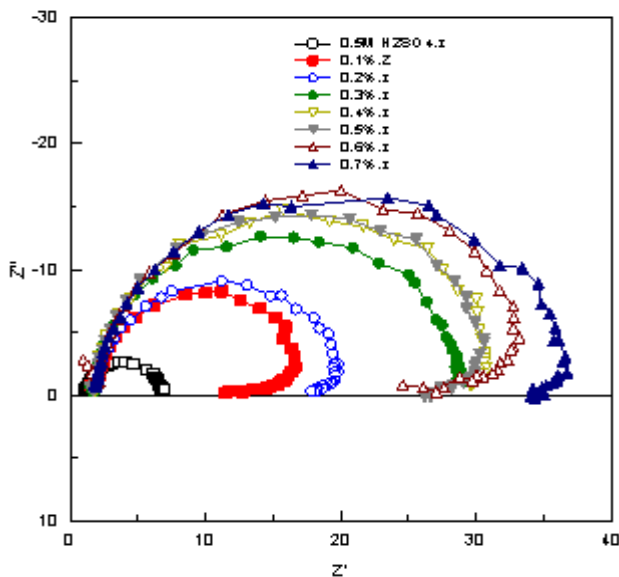


Figure 7a Bode plot of mild steel in the presence of MS/H<sub>2</sub>SO<sub>4</sub> in ACS

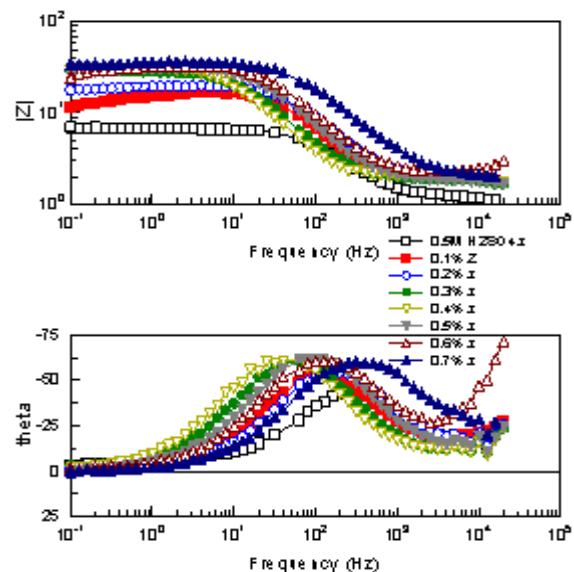


Figure 7b Impedance curve of mild steel in the presence of MS/H<sub>2</sub>SO<sub>4</sub> in ACS

Table 8 R<sub>ct</sub>, C<sub>dl</sub>, and R<sub>p</sub> for MS/HCl in presence of ACS

Sl.no	Conc(%)	R <sub>ct</sub> (Ωcm <sup>2</sup> )	IE(%)	R <sub>p</sub> (Ω/cm <sup>2</sup> )	IE(%)	C <sub>dl</sub> (f/cm <sup>2</sup> )	θ
1	Blank	15.924		2.8567		0.00075484	
2	0.1	21.518	26.0	3.8668	26.1	0.0012007	0.9841
3	0.2	27.396	41.9	4.8993	41.7	0.001309	-0.7260
4	0.3	20.835	23.6	5.0986	44.0	0.0014857	0.9682
5	0.4	29.215	45.5	4.9868	42.7	0.0013552	-0.7953
6	0.5	34.143	53.4	5.0274	43.2	0.00073758	0.0229
7	0.6	63.773	75.0	4.8413	41.0	0.00062532	0.1716
8	0.7	64.324	75.2	4.8008	40.5	0.00061996	0.7748



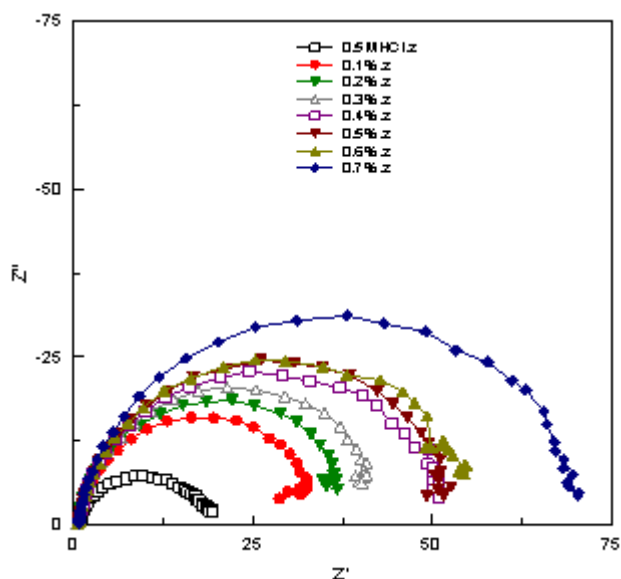


Figure 8a Bode plot of mild steel in the presence of MS/HCl in ACS

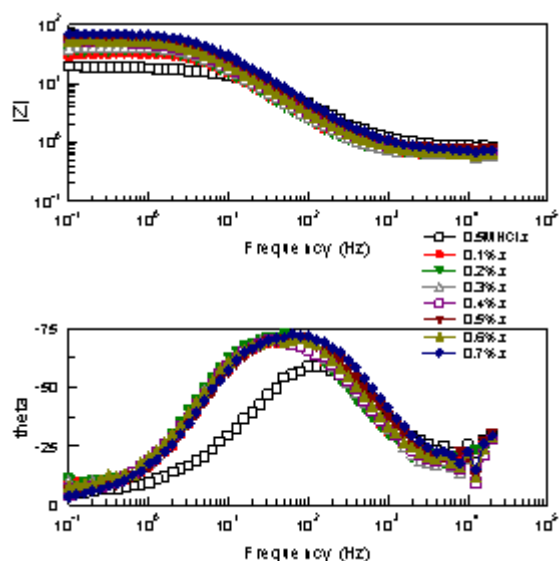


Figure 8b Impedance curve of mild steel in the presence of ACS extract in 1M HCl

**FTIR spectral studies:**

FTIR studies are carried out to characterize the functional groups present in ACS powder. The FTIR frequencies confirmed the presence of components tabulated below (Table 9).

Figure 9 reveals that FTIR spectrum of ACS inhibitor have absorption bands at  $3323\text{ cm}^{-1}$ , and  $2218\text{ cm}^{-1}$  were attributed to O-H stretching [8] and the peaks at  $2339\text{ cm}^{-1}$  and  $2218\text{ cm}^{-1}$  were correspond to C-N stretching. The bands at  $2176\text{ cm}^{-1}$ ,  $2136\text{ cm}^{-1}$ ,  $2011\text{ cm}^{-1}$ ,  $1956\text{ cm}^{-1}$  may be due to strong multiple bonds of alkyne group. The bands at  $1610\text{ cm}^{-1}$  and  $1419\text{ cm}^{-1}$  were attributed to stretching mode of C-C in aromatic and aliphatic ring, a band at  $1253\text{ cm}^{-1}$  may correspond to C-N stretching mode present in aromatic amine. A sharp band at  $1016\text{ cm}^{-1}$  may be due to carbonyl stretching.

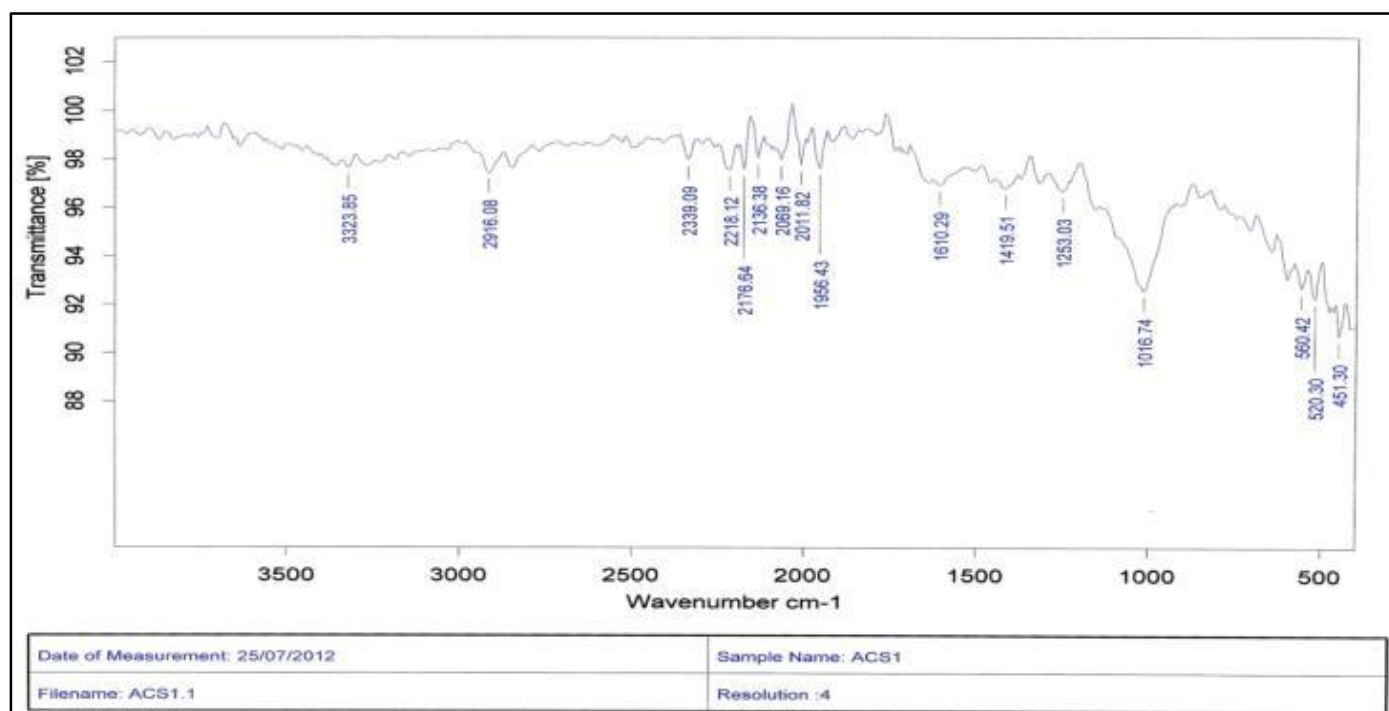


Figure 9 FTIR spectrum of ACS

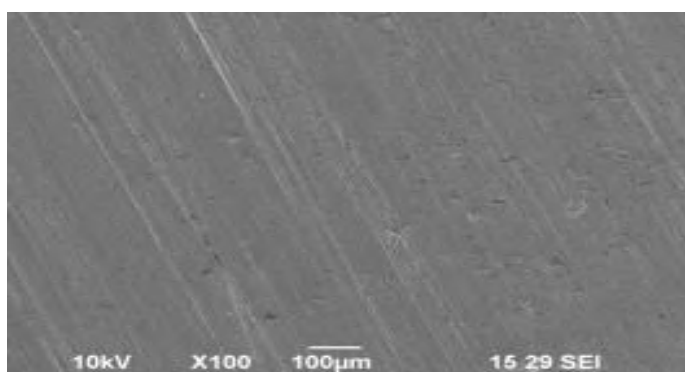


**Table 9 FTIR frequencies for ACS**

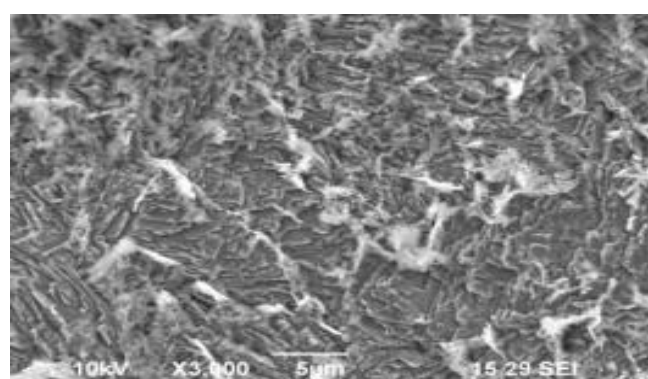
Frequency observed $\text{cm}^{-1}$	Functional group	Compound present
3293	OH – stretching	Arecoline and guvacoline (methyl 1-methyl-1, 2, 3, 6-tetrahydro pyridine-3-carboxylate and methyl 1, 2, 3, 6- tetrahydropyridine-3-carboxylate, arecatannin, gallic acid, a fixed oil gum, a little terpineol, and Arecaidine.
2891	C-H stretching, alkene	
2467	$\equiv\text{CN}$ , C – C alkyne, CHO stretching	
2281, 2212	C=C,CN stretching	
2152,2070, 2028, 1888	$\equiv\text{C}$ C, alkyne stretching	
1620	C=O stretching	
1433	C-C stretching in ring	
1249	C-N aliphatic amine stretching	
1025	=C-H stretching	
815	N-H, carboxylic acid stretching	
642	C- Cl stretching	

### Microstructure Study of mild steel Surface by SEM at 30°C

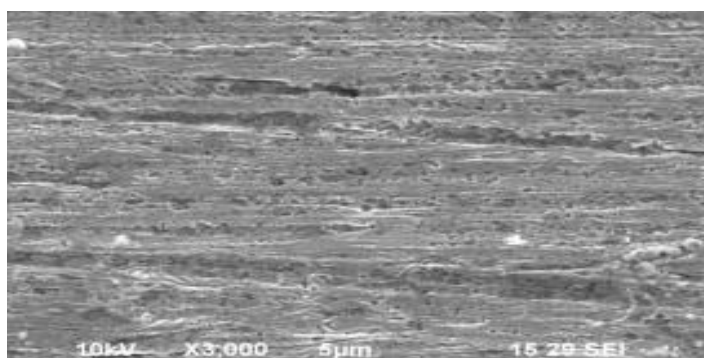
Figure 10a reveals the microstructure of polished MS before placing it in the test solution. The scan shows that a solid and homogeneous surface is found. Figure 10b illustrates the effect of 0.5M HCl on MS sample after 3h immersion at 30°C, it appears that the presence of general corrosion and pitting corrosion (a large number of vacuoles with different sizes). Figure 10c exhibit the effect of 7% concentration of ACS in 1M HCl, it is obvious that the presence of thin porous and protection layer on MS surface contain of numerous pits less than that appears in case of HCl acid alone, the pits are deep and contain a local cell and pitting corrosion which increase the corrosion rate (acceleration). This observation indicated that corrosion rate is reduced to a very low value in the presence of the inhibitor [9]. This might be due to the adsorption of inhibitor molecules on the metal surface as a protective layer.



**Figure 10a SEM photographs for Plain MS**



**Figure 10b SEM photographs for Blank HCl**



**Figure 10c SEM photographs for MS/HCl/ ACS**

**Optical micrograph images:**

. The Optical electron micrographs of the MS in the initial state, after pickling with 0.5M H<sub>2</sub>SO<sub>4</sub> and 1MHCl and in the presence of ACS inhibitor are shown in the Figure 11a to 11c and Figure 12a to 12c respectively. Initial MS micrograph was smooth with grains. Due to the exposure of MS to acid, pits were formed on the surface. In the presence of ACS inhibitor, the pits disappeared. This is due to the presence of adsorbed layer of inhibitor molecules. It can be concluded that corrosion must be inhibited in the presence of inhibitor and hence corrosion was inhibited strongly when the inhibitor is present in acid media.

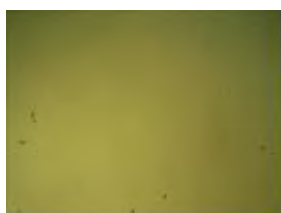


Fig.11a Plain MS



Fig.11b Blank H<sub>2</sub>SO<sub>4</sub>



Fig.11c ACS/ H<sub>2</sub>SO<sub>4</sub>

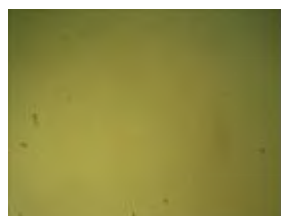


Fig 12a Plain MS



Fig 12b Blank HCl




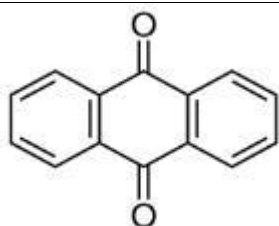
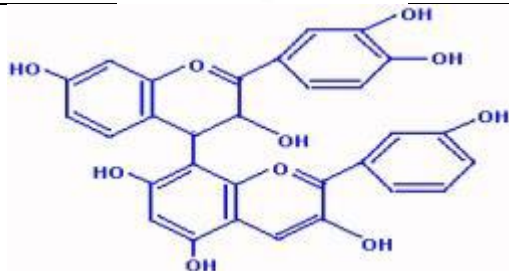
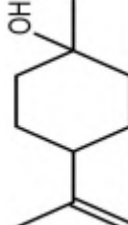
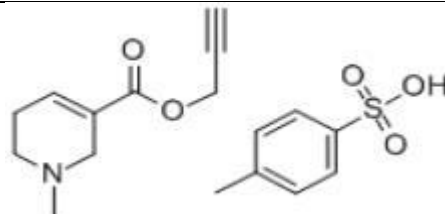
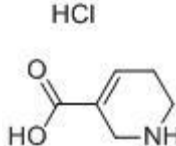
Fig 12c ACS/HCl

**Mechanism of inhibition**

The adsorption of the inhibitor molecules on the mild steel surface is due to the donor acceptor interaction between  $\pi$  electrons of donor atoms N, O and aromatic rings of inhibitors and the vacant d orbitals of iron surface atoms. If no electron transfer occurs, this kind of adsorption is frequently called physisorption. However, during adsorption an electron transfer between the substrate and the adsorbed molecule may occur with orbital overlap between a single pair of electrons of the adsorbed molecule, with N, O and S and empty bands of the metal. Strong interaction is called chemisorption and the adsorption itself is probably the most important type of interaction between the metal surfaces and an inhibitor molecule, In general; organic inhibitors have reactive functional groups, which are the sites for the chemisorption process. The inhibitor molecules are large and flat enough to block more surface areas of the mild steel[10]. The inhibitor molecules can also be adsorbed on the metal surface in the form of negatively charged species which can interact electrostatically with positively charged metal surface. Structure of some compounds present in inhibitor was given in Table 10.

**Table 10 List of phytochemicals and chemicals present in plants**

Name of the compound	Structure
Structure of alkaloids	

Structure of Anthraquinone	
Structure of tannin	
Structure of Beta-Terpineol	
Structure of arecaidinepropargyl ester tosylate	
Guvacine hydrochloride	 HCl

## 5. CONCLUSION:

The investigated inhibitor was proved to be temperature resistance in nature through high temperature studies. Electrochemical studies confirmed the adsorption nature of extract. Mixed mode of inhibition was confirmed by Potentiodynamic studies. SEM and optical microscope analysis were conducted for the conformation of surface corrosion inhibition nature.

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