



# SYNTHESIS, CHARACTERIZATION AND INVESTIGATION OF CHALCONE AS CORROSION INHIBITORS FOR MILD STEEL IN HYDROCHLORIC ACID

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**Keywords:** Corrosion; mild steel; chalcone; corrosion inhibitor; weight loss; acidimetry.

We are reporting a series of chalcone based corrosion inhibitors for mild steel. The compounds were synthesized by grinding azoacetophenone and substituted aromatic aldehyde together in the presence of a base. The compounds were isolated in high purity. The synthesized chalcones' corrosion inhibition activity was studied using weight loss and acidimetric method for mild steel in 1 M hydrochloric acid. The experimental results showed that all corrosion inhibitors' inhibition efficiency increases with an increase in concentration and decreases with an increase in temperature. The good inhibition activity is attributed to the formation of a protective layer on mild steel surfaces. Adsorption of inhibitor on mild steel surface obeyed Langmuir's adsorption isotherm. The mechanical properties of mild steel specimen were also evaluated with Universal Testing Machine (UTM).

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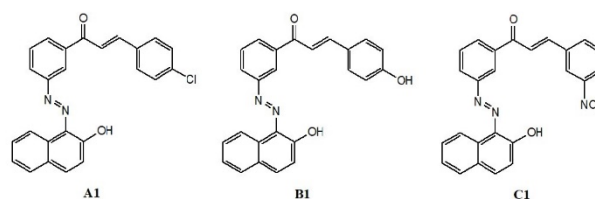
## INTRODUCTION

Mild steel is widely used in various chemical industries like petrochemical, fertilizer, pharmaceutical, etc., due to its easy availability, low cost and high mechanical strength.<sup>1</sup> Mild steel frequently contacts acids, e.g., in acid pickling, descaling, etching, cleaning and oil well acidification, and other applications.<sup>2-4</sup> Besides, mild steel easily undergoes corrosion at different chemical conditions like acidic, alkaline and salt solution. These demand the necessity for the development of excellent corrosion control techniques.

Many methods can control corrosion of mild steel; one of them is the use of corrosion inhibition.<sup>5</sup> Most of the corrosion inhibitors used for the acidic medium are organic compounds containing nitrogen, oxygen and sulfur.<sup>6-9</sup> The prevention of corrosion of metal is due to the adsorption of inhibitor molecules on the surface. The adsorption of inhibitors on the metal surface varies from molecule to molecule. It depends mainly on physico-chemical properties like the size of the molecule, molecular weight, functional group, molecular weight, steric hindrance, the polarity of functional group, electron donor atoms, and electronic structure molecule. Nitrogen, phosphorous, oxygen and sulfur-containing organic inhibitors are very effective corrosion inhibitors in acidic medium.<sup>10,11</sup> Among them, the nitrogen-containing organic inhibitors are effective in hydrochloric acid, while sulfur-containing organic inhibitors are effective in sulphuric acid medium. The inhibition effect of inhibitors for the specific metal depends on the number of atoms contributing to the adsorption process.

The common organic inhibitors contain N, P, S and O, and these atoms take part in the adsorption process. Some studies show that azo containing organic compounds act as a good corrosion inhibitor for metals in acidic condition.<sup>12-15</sup> These motivated us to design and synthesize chalcone derivatives consisting of azo group.

In the present work, we focus on the synthesis of chalcones (A1, B1, C1, Figure 1) containing azo group with other heteroatoms and their application as a corrosion inhibitor in hydrochloric acid for mild steel. We are studying the inhibitory effects of mild steel by the weight-loss method and acidimetric method in 1 M hydrochloric acid.



**Figure 1.** Structure of inhibitors: A1, B1 and C1

## EXPERIMENTAL

### Materials

All the chemicals and solvents for synthesis and test were reagent grade and used without further purification. Mild steel sheets were purchased from a local vendor. Sheet cut into the desired size and all samples were abraded with emery paper range from 600-1200 grade, washed with water, acetone and alcohol, and dried in the open. 1 M HCl solution prepared by properly diluting analytical grade 37 % hydrochloric acid.<sup>16-18</sup>

## Synthesis of inhibitors

In the representative process, to a round bottom flask, 25 ml of HCl and 6.75 g of 3-amino acetophenone were added. After dissolution, the solution is cooled to 5 °C. Keeping the temperature below 5 °C, sodium nitrite solution (3.45 g in 10 ml water) was added to the above solution within 30 minutes. Subsequently, another aqueous solution of 2-naphthol in a sodium hydroxide (7.2 g of 2-naphthol, 2 g of sodium hydroxide and 50 ml of water) solution was added dropwise to the above reaction mixture. The reaction mixture was stirred for 30 minutes at below 5 °C. After completion of the reaction, the products were filtered and washed with water up to neutral pH and dried. The product obtained in this step is azoacetophenone and used for further reaction to obtain chalcones.

In the chalcone formation step, the azoacetophenone (16 mmol) formed in the above step was mixed with substituted benzaldehyde (16 mmol) and sodium hydroxide (16 mmol) in an equal molar ratio in a mortar. The reaction mixture was ground with pastel for 15 minutes in the mortar and allowed to stay as it is for overnight and then washed with water, filtered and dried. The crude product was further purified by crystallization in chloroform.<sup>19-22</sup>

## Characterization of the chalcones

The characterization of all three compounds was done by melting point determination, Infra-red spectroscopy and mass spectrometry. The FT-IR and mass spectra for the compounds are given in the Electronic Supplementary Material. The specification of **A1**, **B1** and **C1** are given below. The main characteristics are the following:

### 3-(4-Chlorophenyl)-1-{3-[(2-hydroxynaphthalen-1-yl)diazenyl]phenyl}prop-2-en-1-one (**A1**).

Orange powder, M.P: 148 °C, FT-IR (KBr, cm<sup>-1</sup>) OH<sub>str</sub> (3610), CH<sub>str</sub>(alkene) (3055), CH<sub>str</sub> (2924), C=O<sub>strc</sub>(conjugated ketone) (1681), OH<sub>bend</sub> (1327), C-N<sub>str</sub> (1157), C-Cl (756), M<sup>+</sup>(m/z)=412, base peak: m/z=143.

### 1-{3-[(2-Hydroxynaphthalen-1-yl)diazenyl]phenyl}-3-(4-hydroxyphenyl)prop-2-en-1-one (**B1**)

Orange powder, M.P: 88 °C, FT-IR (KBr, cm<sup>-1</sup>) OH<sub>str</sub> (3209), CH<sub>str</sub> (alkene) (3063), OH<sub>str</sub>(intramolecular) (2700), C-H bending (1959), C=O<sub>str</sub> (conjugated ketone) (1658), OH bending (1373), C-O<sub>str</sub> (1242), C-N<sub>str</sub>(1149), base peak: m/z=115.

### 1-{3-[(2-Hydroxynaphthalen-1-yl)diazenyl]phenyl}-3-(3-nitrophenyl)prop-2-en-1-one (**C1**)

Orange powder, M.P: 164 °C, FT-IR (KBr, cm<sup>-1</sup>) OH<sub>str</sub> (3556), CH<sub>str</sub>(alkene) (3086), C-H<sub>str</sub>(2870), C=O<sub>str</sub> (conjugated ketone) (1666), N-O<sub>str</sub>(NO<sub>2</sub>)(1519), OH<sub>bending</sub> (1350), C-N<sub>str</sub>(1149), M<sup>+</sup>(m/z)=423, base peak: m/z=144.

## Measurements of corrosion inhibition activity

100 mL 1 M HCl solution was taken in a flask and inserted a mild steel coupon (dimension: L×B×H = 2×5×0.012 cm) into the solution and pH was determined by using a pH meter. The flask was kept for 48 hours at 25 °C without stirring and measured the pH again. The difference of concentration was calculated from the pH change, which directly correlates with the quantity of acid consumed by mild steel during the corrosion process. Based on this, the corrosion rate and inhibition efficiency were calculated. Corrosion inhibition activity was also evaluated in different concentrations of the inhibitor, i.e., 100, 200, 300 and 400 ppm in the acid solution. The concentration of hydrogen ion was determined by pH measurement.<sup>23,24</sup>

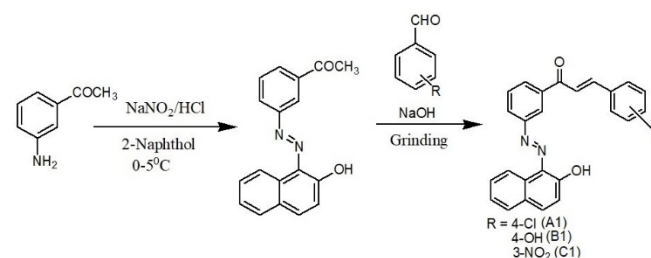
## Tensile strength

For tensile strength measurements, mild steel specimens were treated in acid solution with or without inhibitor. Tensile strength was measured in the Universal Testing Machine (Model: TWI-TUTE-40T). The test specimens were prepared by following the standard method (ASTM A 370) and dimension is shown in Figure 3. The mild steel specimens were properly arranged in the Universal Testing Machine and find out the required force till it reaches the fracture.<sup>25</sup>

## RESULTS AND DISCUSSION

### A general method for synthesis of chalcones

The azoacetophenone compound was mixed with substituted benzaldehyde and sodium hydroxide in an equal molar ratio in a mortar. The reaction mixture was ground with pastel for 15 minutes in the mortar and allowed to stay as it is for overnight.



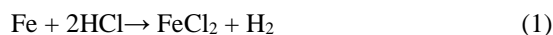
**Figure 2.** Reaction scheme for the preparation of inhibitors **A1**, **B1**, **C1**

### Evaluation of corrosion inhibition activity

#### Acidimetric method

The method is based on pH response to the corrosion reaction with or without the inhibitor in the specific acidic solution (1.0 M HCl). In the typical process, the specific acid solution's pH was determined before and after the insertion of coupons by using the pH meter. As the corrosion progresses, the acid (HCl) is consumed in the reaction by reaction with iron (see below equation), which altered the pH of the medium.

The rate of corrosion was determined by measuring the change in concentration of hydrogen ion, i.e., pH, in the acid solution (1.0 M HCl). Based on the data, the inhibition efficiencies of the chalcones were calculated and are shown in Table 1.



This technique's results are similar to those obtained from the weight loss method (*vide infra*). The two methods of corrosion monitoring gave almost the same values of inhibition efficiency. However, the values of the acidimetric technique were slightly lower than the weight loss method. The corrosion rate of mild steel with and without inhibitors was calculated from the following equation.

$$CR = \frac{[\Delta H^+]}{At} \text{ mol dm}^{-3} \text{ cm}^{-2} \text{ h}^{-1} \quad (2)$$

where

$A$  is the surface area of mild steel coupon in  $\text{cm}^2$ ,

$t$  is the time of insertion in acid solution in hours,

$\Delta H^+$  is the difference of concentration of  $\text{H}^+$  ion.

The inhibition efficiency of corrosion inhibitors was calculated from the following equation.

$$\% IE = 1 - \left( \frac{[\Delta H^+]_{inh}}{[\Delta H^+]_{uninh}} \right) \times 100 \quad (3)$$

Where,  $[\Delta H^+]_{inh}$  and  $[\Delta H^+]_{uninh}$  indicate a change in concentration of hydrogen ion in the presence and absence of inhibitor, respectively.

### Weight loss method

The weight-loss method was performed as described in earlier literature.<sup>26-29</sup> In the typical process, mild steel sheets were cut into  $2 \times 5$  cm and the width was 0.012 cm. The sheets were scrubbed with emery paper (600-1200 grade), washed with water to remove rust and then washed with acetone to degrease and dried. After weighing, the sheets were immersed in 1 M HCl solution with or without inhibitors. All samples were kept in an open beaker for 48 hours. After the immersion period, mild steel sheet coupons were removed from the solution, washed with water, dried and weighed again. Subsequently, all inhibitors with higher efficiency at particular concentrations were evaluated at different temperatures (25, 35, 45, and 55 °C) and calculated the weight loss, corrosion rate, corrosion inhibition and surface coverage according to the below equation.

$$CR = \frac{[\Delta H^+]}{At} \text{ mg cm}^{-2} \text{ h}^{-1} \quad (4)$$

Surface coverage

$$\theta = \frac{W_0 - W_1}{W_0} \quad (5)$$

$$\% IE = \frac{W_0 - W_1}{W_0} \times 100 \quad (6)$$

where

$\Delta w$  is weight loss in mg,

$A$  is the area in  $\text{cm}^2$ ,

$t$  is the time of immersion in hours,

$W_0$  is weight loss without inhibitor and

$W_1$  is weight loss with inhibitor.

**Table 1.** Values of corrosion rate for acidimetric method.

Corrosion rates from acidimetric method			
Inhibitor	Conc., ppm	CR, mol dm <sup>-3</sup> cm <sup>-2</sup> h <sup>-1</sup>	% IE
Blank	-	$1.3 \times 10^{-3}$	-
A1	100	$5.3 \times 10^{-4}$	58.73
	200	$4.4 \times 10^{-4}$	65.94
	300	$3.8 \times 10^{-4}$	70.69
	400	$2.9 \times 10^{-4}$	77.54
B1	100	$4.8 \times 10^{-4}$	62.96
	200	$3.8 \times 10^{-4}$	70.69
	300	$3.3 \times 10^{-4}$	74.03
	400	$2.4 \times 10^{-4}$	81.21
C1	100	$4.6 \times 10^{-4}$	64.43
	200	$3.8 \times 10^{-4}$	70.69
	300	$2.9 \times 10^{-4}$	77.54
	400	$2.0 \times 10^{-4}$	84.08

The weight-loss method is a very common and powerful tool to monitor the corrosion rate of metals. This method gives reliable results. Table 2 shows weight loss measurement results for mild steel in 1 M HCl at different concentrations of the compounds and temperature. The value of the rate of corrosion, inhibition efficiency, and surface coverage was calculated from weight loss data.

The result shows that inhibitors efficiency of all inhibitors viz. A1, B1 and C1 were range from 60.61 to 87.30 %. All inhibitors show the highest inhibition efficiency at a concentration of 400 ppm. Among all, compound C1 shows the highest inhibition efficiency at a concentration of 400 ppm and 25 °C. The higher inhibition efficiency of C1 is attributed to the presence of the  $\text{NO}_2$  group. It was also clear from results that the compounds' inhibition activity increases with an increase in concentration and decreases with the increase of temperature.

### Effect of temperature

To study the effect of temperature on the corrosion inhibition, testing was performed by using different temperatures from 25 to 55 °C with the inhibitors A1, B1 and C1 having a concentration of 400 ppm in 1M hydrochloric acid. In the temperature study, only the weight loss method was used and the results are shown in Table 3.

**Table 2.** Values of corrosion rate, % IE and surface coverage from weight loss method.

Corrosion rate and % IE for weight loss method				
Inhibitor	Conc., ppm	CR, mg cm <sup>-2</sup> h <sup>-1</sup>	% IE	Surface coverage, $\theta$
Blank	-	1.59	-	-
	100	0.62	60.61	0.6061
A1	200	0.51	67.76	0.6776
	300	0.43	72.50	0.7250
	400	0.31	80.30	0.8030
	100	0.54	65.86	0.6586
B1	200	0.41	73.66	0.7366
	300	0.33	78.92	0.7892
	400	0.23	85.41	0.8541
C1	100	0.50	68.19	0.6819
	200	0.39	75.2	0.7520
	300	0.29	81.32	0.8132
	400	0.20	87.30	0.8730

From the results, it has been clear that the corrosion rate increases with the increase in temperature for mild steel in acid solution. The inhibition efficiency decreases with an increase in temperature indicates the desorption process of inhibitor molecules that are already adsorbed on the surface of mild steel. At higher temperatures, the desorption process dominated; hence surface exposure increases resulting in a higher corrosion rate of mild steel. This trend suggests that the inhibitor molecules are adsorbed on a mild steel surface via physical adsorption.

**Table 3.** Values of inhibition efficiency for weight loss method at a different temperature at concentration 400 ppm.

Inhibitor	% Inhibition efficiency for weight loss method			
	Temperature, °C			
	25	35	45	55
A1	80.3	75.9	69.6	63.7
B1	85.4	80.7	74.5	68.8
C1	87.3	81.9	76.9	71.4

To understand effect of temperature on mechanism of inhibition, a kinetic study was done. The activation energy ( $E_a$ ) of the corrosion process can be obtained by using the Arrhenius equation as below.<sup>30,31</sup>

$$\ln CR = \ln A - \frac{E_a}{RT} \quad (7)$$

where

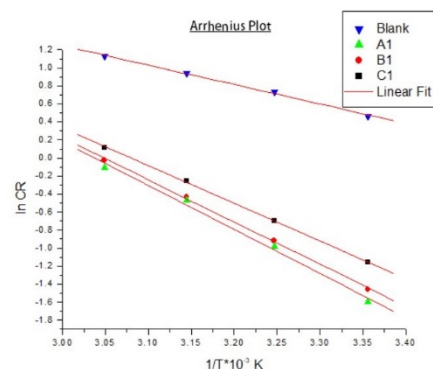
$R$  = gas constant,

$T$  = absolute temperature and

$A$  = pre-exponential factor.

The Arrhenius plot of  $\ln CR$  vs.  $1/T$  for the blank and in the presence of inhibitors is a straight line and the slope of the straight line gave a value of  $E_a$ . The activation energy values calculated from the slope of the straight line are shown in Table 4. The  $E_a$  values of all three inhibitors were higher than that in the absence of inhibitor, which may be related to the occurrence of physical adsorption phenomena through the formation of protective film on the mild steel surface. This also indicates that in the presence of an inhibitor, dissolution

of mild steel in acid becomes more difficult, which results in a decrease of corrosion. The further low value of  $E_a$ , which was under  $80 \text{ kJ mol}^{-1}$ , suggests physical adsorption.

**Figure 3.** Arrhenius plot for mild steel in 1 M HCl with and without inhibitors.

### Adsorption isotherm

The adsorption mechanism of inhibitor is generally explained with the help of adsorption isotherm. Here, the values obtained from the weight loss method were fitted in Langmuir isotherm (equation 8). In particular,  $c/\theta$  was plotted against  $c$  and  $K_{ads}$  was calculated from the intercept. The data are shown in Table 4.

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \quad (8)$$

where

$c$  is the concentration of inhibitor in ppm,

$\theta$  is surface coverage and

$K_{ads}$  is equilibrium constant.

The fitment of experimental data in Langmuir isotherm indicates single layer adsorption.

The standard adsorption free energy ( $\Delta G_{ads}^0$ ) depends on  $K_{ads}$  and can be calculated by using the following equation.

$$\Delta G_{ads}^0 = -RT \ln(55.5 K_{ads}) \quad (9)$$

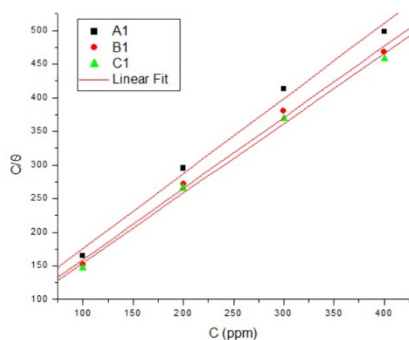
A negative value of  $\Delta G_{ads}^0$  for all three inhibitors indicates the spontaneous adsorption onto the metal surface. In general, if  $\Delta G_{ads}^0 < 20 \text{ kJ mol}^{-1}$ , the inhibitor molecules attract to the metal surfaces through electrostatic attraction, indicating physical adsorption. On the other hand, when  $\Delta G_{ads}^0 > 40 \text{ kJ mol}^{-1}$ , the organic compounds adsorbed on the metal surface through chemical bonds, indicating chemical adsorption.<sup>32-35</sup> All the tested compounds, i.e., A1, B1 and C1 possess  $\Delta G_{ads}^0$  values below  $20 \text{ kJ mol}^{-1}$ .

### Tensile strength

A total of four specimens of mild steel was tested and measured the tensile strength and yield strength. The experimental data are tabulated in Table 5.

**Table 4.** Thermodynamic parameter for inhibitors at concentration 400 ppm.

Inhibitor	$K_{ads}$	$\Delta G^0_{ads}$ , kJ mol <sup>-1</sup>	$E_a$ , kJ mol <sup>-1</sup>
Blank	-	-	17.84
A1	6351	-14.48	34.6
B1	5324	-14.59	38.84
C1	5058	-13.93	40.5

**Figure 4.** Langmuir adsorption isotherm for mild steel in 1 M HCl solution with different concentrations of inhibitors.

The results did not show any significant difference in tensile strength and yield strength between the inhibited and uninhibited mild steel specimen. It has been already proved<sup>36</sup> that corrosion reduces the tensile strength of metal but gradually. While the application of inhibitors in acid solution is mainly for acid pickling and that process does not require a long duration. Thus, it is difficult to establish a correlation between the effect of the inhibitor on the test specimens and their tensile strength.

**Table 5.** Test result for tensile strength

Inhibitor	Tensile strength	Yield strength,
	N mm <sup>-2</sup>	
Blank	484.54	323.91
A1	483.69	325.11
B1	487.51	322.97
C1	488.00	325.87

## CONCLUSION

In conclusion, a series of chalcones were successfully synthesized, isolated and characterized by different analytical techniques. The chalcones were investigated for their corrosion inhibition activity for mild steel in 1M HCl. The notable features of the compounds are as follows.

All three chalcones were good inhibitors for mild steel in 1 M HCl.

Among the three inhibitors, C1 showed the highest inhibition efficiency.

The inhibition efficiency of all inhibitors was increased with an increase in the concentration of inhibitors.

The inhibition efficiency of all inhibitors was decreased with an increase in temperature.

Adsorption of inhibitors on mild steel surface is due to physical adsorption.

The adsorption process of inhibitors on mild steel surface obeyed Langmuir's isotherm.

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