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# Corrosion inhibition behavior of nonionic surfactant based on amino acid on mild steel in acid media

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## ABSTRACT

Amino acid based surfactant N-alkyl cysteine, designated as ( $C_{12}Cys$ ) was synthesized and purified. The chemical structure of the synthesized surfactant was confirmed by FT-IR, <sup>1</sup>H-NMR and elemental analysis. The inhibition efficiency of the prepared inhibitor on mild steel in 1M HCl was studied using potentiodynamic polarization, and electrochemical impedance spectroscopy. The synthesized compounds act as mixed type inhibitor and adsorb on MS surface in accordance with Langmuir adsorption isotherm. The EIS results revealed a greater charge transfer resistance in ( $C_{12}Cys$ ) solution compared to that in blank solution. The AFM results exhibited a lowering in the roughness of corroded MS surface in presence of ( $C_{12}Cys$ ) surfactant. Correlation between the guantum chemical calculations and inhibition efficiency was discussed.

Keywords: Acid Corrosion; Corrosion inhibitor; Amino acid based surfactant; EIS; DFT calculation

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#### INTRODUCTION

Mild Steel (MS) is highly acceptable by diverse industries such as petro-chemical industry, oil and gas industry due to its high strength, malleability, low cost, and affordability [1]. Due to these properties there is a desire to maximize the lifetimes of MS items, with one of the main causes of degradation being corrosion, specifically in aggressive acidic environments [2].

Acid is used to pickle steel. HCl is more expensive than H<sub>2</sub>SO<sub>4</sub>, but it pickles much faster while minimizing base metal loss. In order to prevent the base metal attack during these processes, various approaches are used to minimize corrosion, with inhibitors being one of the cheapest and familiar methods [3,4]. Compounds having N, S, and O atoms with high electron density have been found to be potential inhibitors against metal corrosion in many environments [5-7]. But many of them are highly toxic and raise environmental concerns. In the current time, the principal objective in selecting corrosion inhibitors for practical application apart from efficiency is the eco-friendliness of the inhibitor because of problems of environmental pollution. The advantages of using surfactants as corrosion inhibitors are its high inhibition efficiency, low cost, low toxicity and easy production [7,8].

Within this work, we provide a complete view of amino acid-based surfactant derived from cysteine, designated as ( $C_{12}Cys$ ), where C represent the no. of C atoms in hydrophobic chain moiety, from the viewpoint of synthesis and corrosion inhibition performance. The corrosion inhibition performance was investigated by potentiodynamic polarization measurement, electrochemical impedance measurement, and AFM. Furthermore, the inhibition performance of the studied inhibitor was correlated with quantum-chemical calculations in anticipation that the correlation will be helpful in the design and synthesis of new inhibitors with higher inhibition efficiency.

#### EXPERIMENTAL PROCEDURE

#### SPECIMEN COMPOSITION AND SOLUTION

Rectangular specimens were prepared from commercially available MS rods of chemical composition (wt %): C-0.061, Mn-0.181, P-0.018, Cr-0.035, Mo-0.054, Al-0.017, V-0.034 and Fe-99.59. Prior to each experiment, the surface of the MS specimens was hand polished with mechanical grade emery papers of 320–1200 grade. Before using the specimen in electrochemical measurements as the working electrode, it was well cleaned, degreased with ethanol and washed thoroughly with double distilled water.

1 M HCl solution was prepared by diluting analytical grade HCl (37%) with double-distilled water. For corrosion measurements, the concentration range of the synthesized inhibitor varied from  $1 \times 10^{-3}$  to 0.2 mM. Double-distilled water was used for preparing the test solutions in all measurements.

#### **CHEMICAL SYNTHESIS OF INHIBITOR**

 $(C_{12}Cys)$  used in this work was synthesized and purified as reported [9]. The chemical synthesis of the compounds is shown in Fig 1. And spectral data is given below



Figure 1: Pathway for the synthesis of (C<sub>12</sub>Cys) surfactant

## N-alkyl cysteine monomeric surfactant, (C<sub>12</sub>Cys)

FT-IR (KBr) (cm<sup>-1</sup>): 3421 (NH), 2921, 2853 (C-H), 2552 (S-H), 1599 (C=O), 1470 (O-H) 1056 (C-N), 719 (-CH<sub>2</sub>)<sub>n</sub>- skeletal.

<sup>1</sup>H-NMR (DMSO, 500MHz): δ 0.87 (t, 6H), 1.23-1.29 (m, 18H), 1.49-1.51 (m, 2H), 2.50-2.55 (m, 2H) 2.64 (m, 2H), 3.64 (m, 1H).

Elemental analysis: Calcd. (%) C-62.28; H-10.72; N-4.89 Found (%): C-61.16; H-10.39; N-4.98.

## **ELECTROCHEMICAL MEASUREMENTS**

The electrochemical measurements were done in an AUTOLAB 1L corrosion cell (three neck) that have Ag/AgCl electrode, provided with a luggin capillary probe as reference electrode along with the Pt foil as counter electrode. MS coupon, which acted as working electrode, was attached to a specially designed holder. In order to minimize IR drop the tip of luggin probe was made very close to the surface of working electrode. The measurements were accomplished by an Autolab potentiostat/galvanostat, Model 128N with inbuilt impedance analyser FRA 2 at 303K under unstirred condition. Prior to the commencement of an experiment, the potential was continuously monitored until it was stabilized and a steady state open circuit potential (OCP) was achieved.

For anodic and cathodic polarization curves, an over potential voltage of -250 mV vs. Ag/AgCl to +250 mV vs. Ag/AgCl with reference to OCP was applied at a scan rate of 0.001 V/s. Corrosion current density ( $I_{corr}$ ), equilibrium corrosion potential ( $E_{corr}$ ), anodic Tafel slope ( $\beta$ a) and cathodic Tafel slope ( $\beta$ c) values were obtained using NOVA 1.11 software. Inhibition efficiency ( $_{PDP}$ ) and surface coverage ( $\theta$ ) at different inhibitor concentrations were calculated by the Eqs. given below, where  $I_{corr}$  and  $I^{(i)}_{corr}$  are the corrosion current densities without and with inhibitor, respectively.

 $\eta_{PDP}$  (%) =  $\theta \times 100$ 

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$$\theta = \frac{I_{\rm corr} - I^{(i)}_{\rm corr}}{I_{\rm corr}}$$
(2)

Impedance spectra were obtained at OCP within the frequency domain of  $10^{-2}$  to  $10^{5}$  Hz by applying 10 mV sine wave AC voltage. Before starting experiments, the electrochemical system was stabilized to gather EIS data at OCP. The obtained EIS spectra were analyzed for the values of  $R_{ct}$ , the charge transfer resistance and the  $C_{cll}$ , the double layer capacitance. Inhibition efficiency ( EIS) was computed at each inhibitor concentration with the help of Eq.

$$\eta_{R}(\%) = \frac{R_{ct}^{(i)} - R_{ct}}{R_{ct}^{(i)}}$$
(3)

where, *R*<sub>ct</sub> and *R*<sub>ct</sub><sup>(i)</sup> are the charge transfer resistances without and with surfactant, respectively.

#### ATOMIC FORCE MICROSCOPY (AFM)

For AFM analysis the MS coupons of size 2.5 2.0 0.1 cm were prepared, this was followed by their immersion in the test solution without and with optimum concentration of inhibitors for 6 h. After completion of immersion the specimens were retrieved, corrosion products removed mechanically and thoroughly rinsed with distilled water, dried and subjected to AFM analysis. The AFM analysis was made at room temperature in tapping mode in air using AFM-Dimension icon ScanAsyst equipped with Nanoscope V. The scan rate and scanning area were 0.4 Hz and 50 50  $\mu$ m, respectively.

#### QUANTUM CHEMICAL CALCUALTION

Quantum chemical calculation was done using the Density functional theory implemented in the Spartan'14 v1.1.8 software. The exchange–correlation was treated using hybrid, B3LYP functional. A full optimization was performed using the 6-31G (d,p) basis sets. Molecular properties relevant to the activity of the molecules as corrosion inhibitors such as energy of the highest occupied orbitals ( $E_{HOMO}$ ), energy of unoccupied molecular orbitals ( $E_{LUMO}$ ), energy gap ( $\Delta E$ ), were obtained.

#### RESULTS

#### POTENTIODYNAMIC POLARIZATION (PDP)

Anodic and cathodic polarization curves of MS in 1 M HCl solutions in the absence and presence of various concentrations of ( $C_{12}Cys$ ) inhibitor are shown in Fig. 2.

According to the literature a shift in  $E_{corr} > 85 \text{ mV}$  vs. Ag/AgCl with respect to the  $E_{corr}$  of blank solution can be classified as cathodic or anodic type inhibition; otherwise ( $E_{corr} < 85 \text{ mV}$  vs. Ag/AgCl)

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the inhibitor is classified as mixed type inhibition [10]. In the present work, the shift in  $E_{corr}$  values of the inhibited systems compared to the acid solution is < 85 mV, suggesting that the studied inhibitor is a mixed type inhibitor. The anodic (\_\_a) and cathodic (\_\_c) Tafel slopes of (C<sub>12</sub>Cys) changed with inhibitor concentration indicating that these inhibitor controlled both anodic as well as cathodic reactions. All the values of  $\beta_c$  are larger than those of  $\beta_a$  in the tested media, which indicated that the corrosion process is controlled by the cathodic reaction, though the variation of the Tafel slopes with concentrations does not follow a definite trend.

It is also apparent in the Table 1 that the values of corrosion current density ( $I_{corr}$ ) of the inhibited acid solutions containing ( $C_{12}Cys$ ) are significantly smaller compared to those of the free acid solution. This indicates the inhibition of MS corrosion in 1 M HCl solution by the inhibitor. The values of  $I_{corr}$  of the lowest studied concentration of ( $C_{12}Cys$ ) (1  $10^{-3}$ mM) is 0.48  $10^{-4}$  A.cm<sup>-2</sup>, corresponding to an inhibition efficiency of 65.3%. When the concentration was increased from 1  $10^{-3}$  mM to 0.2 mM the  $I_{corr}$  values changed to 0.17  $10^{-4}$  A.cm<sup>-2</sup>, and the inhibition efficiency increased to 86.5%.

C <sub>inh</sub> (mM)	E <sub>corr</sub> (V vs. Ag/AgCl)	a/(V dec⁻ ¹)	c/(V dec⁻ ¹)	<i>I</i> <sub>corr</sub> 10 <sup>-4</sup> (A cm <sup>-2</sup> )	ррр (%)
0	-0.436	0.137	0.135	1.4 0.09	
1 10 <sup>-3</sup>	-0.443	0.103	0.074	0.48 0.04	65.3
7 10 <sup>-3</sup>	-0.420	0.087	0.052	0.35 0.01	74.4
1 10 <sup>-2</sup>	-0.414	0.111	0.088	0.27 0.03	78.7
7 10 <sup>-2</sup>	-0.421	0.086	0.091	0.24 0.03	81.3
0.15	-0.417	0.062	0.063	0.19 0.02	85.6
0.2	-0.435	0.043	0.056	0.17 0.05	86.5

Table 1: PDP Parameters for MS in 1M HCI in the absence and	presence of different
concentrations of (C <sub>12</sub> Cys) (temperature 303	2 K)



Figure 2: PDP curves of MS in 1M HCl solution without and with different concentrations of (C<sub>12</sub>Cys) (temperature 303 2 K)

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#### **ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY (EIS)**

The impedance spectra recorded in the absence and in the presence of various concentrations of studied inhibitor in 1 M HCl solution are shown in Fig. 3(a-b). The impedance parameters were calculated utilizing the application of the equivalent circuit model. The simplest fitting is represented by the Randles equivalent circuit (Fig. 3c), which is a parallel combination of the charge-transfer resistance ( $R_{ct}$ ) and the double-layer capacitance ( $C_{dl}$ ), both in series with the solution resistance ( $R_s$ ). It is clear from the Fig. 3(a) that all impedance spectra exhibit one single capacitive loop, which suggests that the corrosion of MS in 1 M HCl with and without inhibitors is mostly controlled by charge transfer process [11]. Moreover, these diagrams have a similar shape for all tested concentrations, indicating that there is quite no change in the corrosion of interfacial impedance [12]. This capacitance dispersion at the solid surfaces is mainly caused by surface roughness, the chemical heterogeneity of surface, and adsorption–desorption process of inhibitive molecules on MS surface [13]. To overcome this situation, a constant phase element (*CPE*) which corresponds to surface roughness, impurities, degree of polycrystallinity, and adsorption of inhibitive molecules [14] is introduced in the equivalent circuit in place of  $C_{dl}$ .

Impedance function of CPE is defined as follows [15]

$$Z_{CPE} = \frac{1}{Y_0 (j\omega)^n}$$
(4)

where  $Y_0$  is *CPE* constant, *j* is the imaginary unit ,  $\omega$  is the angular frequency and *n* is *CPE* exponent (represents the deviation from the ideal behavior and it lies between 0-1). The lower value of *n* (Table 3) for MS in the studied corrosive solution indicates surface irregularity resulted from metal surface roughening due to corrosion. However, in the presence of the (C<sub>12</sub>Cys), *n* values were found to be increased, suggesting reduction inhomogeneity of surface due to the adsorption of studied inhibitor molecules [16].

$$Y_0$$
 is converted into  $C_{dl}$  by the use of following Eq. [14]  
 $C_{dl} = Y_0 (\omega_{max})^{n-1}$  (5)

where  $m_{max} = 2\pi f_{max}$  ( $f_{max}$  denotes maximum frequency at which the imaginary component of the impedance has a maximum).

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Figure 3: (a) Nyquist (b) Bode plots of MS in 1 M HCl solution without and with different concentrations of inhibitor (c) Randles equivalent circuit used to fit the impedance data

It is apparent from the Table 2 that the  $R_{ct}$  values of the inhibited solutions increased with the inhibitors concentration. This is due to the protective film formed at the MS-acid interface. The  $C_{dl}$  values were decreased with an increase in concentration of ( $C_{12}Cys$ ). The decrease in  $C_{dl}$  value can be attributed to the increase in the thickness of the electrical double layer and/or a drop in the local dielectric constant [16]. Further the decrease in the  $C_{dl}$  values is caused by the replacement of  $H_2O$  molecules by the adsorption of the ( $C_{12}Cys$ ) molecules on the metal surface, leading to a reduction in the number of active sites required for the corrosion reaction. Also, the increase of  $R_{ct}$  with rise in the inhibitor concentration, indicate that the charge transfer process is mainly controlling the corrosion process. In contrast, the better protection provided by an inhibitor can be associated with a decrease in the capacitance of the metal.

Table 2: EIS parameters for MS in 1M HCI in the absence and	presence of different
concentrations of (C <sub>12</sub> Cys) (temperature 303	2 K)

C <sub>inh</sub> (mM)	<i>R</i> <sub>s</sub> ( cm <sup>2</sup> )	$R_{\rm ct}$ ( cm <sup>2</sup> )	CPE		C <sub>dl</sub> 10 <sup>-4</sup> (μFcm <sup>-2</sup> )	EIS (%)
			Y₀ 10 <sup>-4</sup> ( <sup>-1</sup> s <sup>n</sup> cm <sup>-2</sup> )	n		
0	4.64	91.7 5.50	1.51	0.9940	1.36	
1 10 <sup>-3</sup>	1.16	266.8 9.60	1.64	0.9964	1.62	65.6
7 10 <sup>-3</sup>	4.84	342.0 5.47	1.18	0.9955	1.15	73.2
1 10 <sup>-2</sup>	2.04	418.1 11.95	0.89	0.9963	0.88	78.1
7 10-2	5.36	443.1 25.96	0.83	0.9969	0.82	79.3
0.15	1.01	616.3 22.18	0.73	0.9955	0.71	85.1
0.2	8.26	752.9 34.63	0.68	0.9944	0.67	87.8

Bode plots for MS immersed in 1 M HCl solution with and without different concentrations of ( $C_{12}Cys$ ) inhibitor exhibit one time constant, thus showing that the inhibitor systems behaves as a monolayer formation and the dissolution process is controlled by a charge transfer reaction, taking place at the

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steel/solution interface [15]. As we can see in Fig. 3b that increasing the concentration of ( $C_{12}Cys$ ) compound in the investigated solution results in closer to 90 of the phase angle indicating excellent inhibitive behavior due to adsorption of more ( $C_{12}Cys$ ) molecules on the steel surface at higher concentrations. The continuous increase in the phase angle shift in the presence of the inhibitor was obviously associated with the growth of inhibitor film and with the increase in surface coverage on the MS surface, resulting in higher inhibitor is larger than in its absence and the value of impedance was increased with increasing the inhibitor concentrations. This means that the corrosion rate is decreased by addition of ( $C_{12}Cys$ ) compound and continued to decreasing upon increasing the concentration of the inhibitor.

#### ADSORPTION ISOTHERM

The adsorption isotherm provides the nature of interaction between the inhibitor molecule and the metal surface. Experimental values obtained from electrochemical measurement are investigated graphically for fitting several kinds of adsorption isotherm. Among the above isotherms, excellent fitting of the experimental values has been observed by the simplest Langmuir adsorption isotherm model. According to the Langmuir adsorption isotherm, the degree of surface coverage  $\theta$  is related to the concentration of the inhibitor ( $C_{inh}$ ) by the following Eq:

#### $C_{\rm inh}/\theta = 1/K_{\rm ads} + C_{\rm inh}$

(6)

where ' $K_{ads}$ ' stands for the equilibrium constant in the adsorption process. A linear relationship between  $C_{inh}/\theta$  vs.  $C_{inh}$  has been observed with strong correlation coefficient ( $R^2$ ). In Fig. 4, the values of  $R^2$  for the Langmuir adsorption model are close to 1. Adsorption of inhibitor molecules on the adsorbent is of monolayer in nature which is confirmed from the Langmuir adsorption isotherm. High values of  $K_{ads}$  (Table 3) suggest stronger adsorption of the inhibitor molecule on the metal surface and hence better inhibition efficiency [17].



## Figure 4: Langmuir adsorption isotherm plot for MS in 1M HCl solution containing various concentrations of (C<sub>12</sub>Cys)

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The standard free energy of the inhibitor adsorption process,  $\Delta G^{0}_{ads}$ , is related to the adsorption equilibrium constant,  $K_{ads}$ , by the following Eq:

$$K_{ads} = \left(\frac{1}{55.5}\right) \exp\left(\frac{-\Delta G^0_{ads}}{RT}\right)$$
(7)

where 55.5 is the molar concentration of water in the solution expressed in M (mol L<sup>-1</sup>); R is the gas constant (8.314 J K<sup>-1</sup> mol<sup>-1</sup>); and T is the absolute temperature (K). The negative values of  $\Delta G^{0}_{ads}$ (Table 3) ensure the spontaneity of the adsorption process and stability of the adsorbed layer on the steel surface. The  $\Delta G^{0}_{ads}$  values range between -27.52 to -25.77 KJ mol<sup>-1</sup>, (Table 3); this specifies that the adsorption of (C12Cvs) molecules on the MS surface involves physisorption along with chemisorptions [17].

Table 3: Thermodynamic parameters of adsorption for MS in 1M HCI				
Techniques	Slope	<i>R</i> <sup>2</sup>	<b>K</b> ads	G ads (KJ mol <sup>-1</sup> )
PDP	1.15	0.999	1000	-27.52
EIS	1.14	0.998	500	-25.77

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#### **AFM ANALYSES**

AFM analysis was also carried out to investigate the average surface roughness in the polished MS specimen and in the absence and presence of the studied inhibitor in 1 M HCI (Fig. 4). The average surface roughness of polished surface found is 70.8 nm (Fig. 4A). In the absence of (C12Cvs) the surface displayed an extremely rough topography due to an unhindered corrosion attack (Fig. 4B) and the average surface roughness increased from 65.2 nm to 627 nm. In the presence of (C12Cys) the MS shows a smoother surface (Fig. 4C) and the average surface roughness value decreased from 627 to 290 nm for ( $C_{12}Cys$ ).



Figure 4: AFM images of MS after 6 h immersion in 1M HCl solution: (A) Polished MS prior to immersion, (B) uninhibited solution, (C) acid solution with 0.2 mM (C<sub>12</sub>Cys) NIGIS \* CORCON 2017 \* 17-20 September \* Mumbai, India

#### QUANTUM CHEMICAL CALCULATION

In order to correlate the molecular structure of amino acid based surfactant with its inhibition efficiency, quantum chemical calculations were performed. The optimized molecular structures and graphic surfaces of HOMO and LUMO orbitals of ( $C_{12}Cys$ ) are given in Fig. 5. Table 4 lists the quantum chemical parameters such as  $E_{HOMO}$ ,  $E_{LUMO}$ , and E recorded for ( $C_{12}Cys$ ).

The HOMO is the orbital from where electrons are donated to the vacant d-orbitals of the metal to form a coordinate bond, while the LUMO is the orbital where back donation of electrons coming from the metallic surface is accommodated [18]. The higher is the  $E_{HOMO}$ ; the better will be the donating capability of the inhibitor. The energy of the lowest unoccupied molecular orbital indicates the ability of the molecule to accept electrons. Lower the value of  $E_{LUMO}$ , the more probable the molecule would accept electrons. Consequently, the difference between  $E_{HOMO}$  and  $E_{LUMO}$ , referred as the energy gap (E), which is also an important parameter to determine the inhibition efficiency of the inhibitor. Concerning the value of the energy gap  $\Delta E$ , larger values of the energy difference will provide low reactivity to a chemical species. Lesser the values of the  $\Delta E$ , greater will be good inhibition efficiency, because the energy required to remove an electron from the lowest occupied orbital will be low.



Figure 5: (a) Optimized structure, (b) HOMO and (c) LUMO energies of (C<sub>12</sub>Cys)

## Table 4: Quantum chemical parameters for protonated form of (C<sub>12</sub>Cys) calculated using density functional theory at B3LYP/6-31G (d, p) level of theory

<i>Е</i> номо (eV)	E <sub>LUMO</sub> (eV)	*Δ <i>E</i> (eV)	
-9.48	-4.71	4.77	

#### CONCLUSIONS

The amino acid based surfactant referred as  $(C_{12}Cys)$ , was synthesized and characterized. Compound acted as good inhibitor in preventing corrosion of MS steel in 1M HCl solution. Potentiodynamic polarization study showed that  $(C_{12}Cys)$  act as mixed-type inhibitor in acidic solution, diminishing both anodic and cathodic corrosion. An increase in  $(C_{12}Cys)$  concentration resulted in an increase in inhibition efficiencies. High inhibition efficiencies exhibited by the inhibitor is attributed to their adherent adsorption and the adsorption fits Langmuir's thermodynamic model.

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AFM images are suggestive of smoother MS surface for (C<sub>12</sub>Cys) inhibited acid solution compared to uninhibited system. Experimental findings are adequately supported by quantum chemical calculations.

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