



Corrosion inhibition performance of Coriander Seeds extract molecules for pure Aluminum in Hydrochloric acid Medium: A combined Experimental and Quantum chemical approach

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Abstract

The study of inhibitive properties of coriander seeds extract (CSE) molecules over Aluminum surface in 1M hydrochloric acid (HCl) medium was performed by standard weight loss (gravimetric) and electrochemical impedance methods. Result from gravimetric measurements showed that behavior of inhibition efficiency is directly proportional to concentration of inhibitor. Impedance measurement reveals that charge transfer resistance and constant phase element values increases and decreases respectively with increase in concentration of inhibitor. The study indicates that the molecules present in the extract forms a protective layer at metal/solution interface. In addition quantum chemical study was also performed using density functional theory (DFT) to elucidate the inhibitory activity of individual molecule present in the extract. The result from quantum study showed that 10, 12-Hexadecadien-1-ol had the maximum power of adsorption at Aluminum surface to prevent it from corrosion.

Keywords: Corrosion inhibition, coriander seed extract, gravimetric method, electrochemical impedance spectroscopy, HCl, DFT.

Introduction

Extensive corrosion is known to occur due to aggregation of acid on metallic surface leading to its dissolution. Acids found application in industrial acid cleaning, acid pickling and electrochemical etching of Aluminum (Al) foil capacitors which causes severe corrosion of metals¹. Among various acids such as nitric, hydrochloric (HCl) and sulphuric acids, HCl is commonly used for pickling and etching purposes due to its low cost as compared to other acids. The corrosion rate of metals can be reduced with the application of inhibitors in aggressive environment^{2,3}. The organic inhibitor is said to be effective if there is a surplus of electrons (hetero-atoms and π -electron cloud) in its structure which it can donate to metal empty orbital leading to formation of protective layer⁴⁻⁷. There are several factors such as chemical structure of inhibitor, type of metal and aggressive environment which affect the efficiency of an inhibitor⁸⁻¹¹.

The extent of Al corrosion differs with change in corrosive environment and hence the inhibitor should be capable of performing multiple functions. Therefore, combination of inhibitors can be necessary in providing multiple services required in effective corrosion inhibition. In addition, high toxicity coupled with high cost of synthetic organic inhibitors provide sufficient motivational path to researchers in designing environment friendly inhibitors. Therefore, investigation in green corrosion inhibitors continues to proceed and the extract

of various plants were studied for their inhibitive properties due to presence of abundant phytochemicals in them. Some previously reported research shows that piper guineense¹², Launaea nudicaulis¹³, bamboo leaf¹⁴, coriander^{15, 16}, curcuma longa¹⁷ act as corrosion inhibitor for steel. Anethum graveolens L. oil¹⁸, natural honey, vanillin, and tapioca starch¹⁹, Solanum trilobatum²⁰ shows good inhibitory characteristics for Al. However, work on coriander²¹ seeds on Al for their inhibitive performances in phosphoric acid and alkaline medium were previously performed by Prabhu and Rao in 2013. There is no reported work till date can be found on coriander seeds as inhibitor for Al in HCl medium.

In the current research work, the effect of coriander seeds extract (CSE) as corrosion inhibitor for Al in HCl medium has been examined using experimental techniques: gravimetric and electrochemical impedance spectroscopy (EIS). The experimental methods are used for monitoring the inhibition efficiency of CSE. The complex composition of extract makes it difficult to determine the contribution of all the molecules present in it. To overcome the problem, quantum chemical study is performed to understand the overall importance of CSE molecules. The approach involves understanding the presence of highest occupied and lowest unoccupied molecular orbitals of major CSE chemical components and also their individual importance in inhibitory effect using DFT technique.

Material and Methods

The pure Al specimen with composition Al (99.2%), Fe (0.498%), Si (0.144%), Mn (0.031%), Cu (0.007%), Mg (0.005%) and Zn (0.001%) and dimension $3.0 \times 6.0 \times 0.05$ cm was used for the gravimetric measurements. The specimens were abraded using emery paper, degreased with acetone, dried, and weighed before using for gravimetric measurements. Measurements were carried out for Al specimen in 230 ml of 1M acid (HCl) solution and along with various concentration (0.013 – 0.522 g/l) of inhibitor for exposure period of 60 minutes at 35°C.

In electrochemical impedance spectroscopy, Al, platinum and calomel were employed as working, counter and reference electrode. Al and platinum electrode was of circular design with exposed area of 6.156 cm^2 to HCl solution. AUTOLAB instrument was used for electrochemical impedance measurements. Before measurements, electrodes were dipped in aggressive solution for 30 minutes. The potential applied in the form of sine wave across the cell. The frequency applied varies from 10 kHz to 1Hz with amplitude of 10mV. Frequency response analyzer (FRA) was used for analyzing the EIS data obtained from the measurement.

GC-MS analysis was performed for understanding the chemical

composition of extract molecules using GC-MS QP-2010 plus of Shimadzu. GC column used for the analysis is Rtx-5 of 30m X 0.25 mm X 0.25 μm size. The extract components were separated using GC column and detection of the components was performed using mass spectrometer (MS) detector in full scan mode and all the components with mass is to charge ratio (m/z) ranging from 40 to 650 were recognized.

Quantum studies were executed using DFT approach with B3LYP hybrid functional and 6-31 G (d) as a basis set. DFT was employed for elucidating electronic structures and reactivity of molecules present in the extract. The structure optimization of the extract molecules were performed using Gaussian 09 program.

Results and Discussion

Characterization of CSE: CSE molecules obtained from GC-MS analysis along with their percentage area are shown in table-1. The main chemical constituents are: 3,7-dimethyl-1,6-Octadien-3-ol (linalool: 4.06%), 3,7-dimethylacetate-2,6-octadien-1-ol (ODDA: 4.99%), Tetradecanoic acid (TDA: 7.07%), n-Hexadecanoic acid (HA: 18.68%), 6- 10,12-Hexadecadien-1-ol (HDO: 4.25%), 6-Octadecenoic acid, (Z)- (OA: 8.38%), Hexadecanoic acid, tetradecyl ester (HATE: 4.76%), Hexadecadienoic acid, methyl ester (HAME: 4.95%).

Table-1
GC-MS analysis result of CSE

Peak	R. Time (minutes)	Area%	Name of the compound
1.	12.055	4.060	1,6-Octadien-3-ol, 3,7-dimethyl- (linalool)
2.	13.555	2.630	1,5-anhydro-6-deoxyhexo-2,3-diulose (ADD)
3.	20.364	4.990	2,6-octadien-1-ol, 3,7-dimethyl-, acetate (ODDA)
4.	29.454	7.070	Tetradecanoic acid (TDA)
5.	30.277	2.470	2-Tridecenoic acid, (E)- (TA)
6.	33.519	18.680	Hexadecanoic acid <n-> (HA)
7.	35.994	2.010	6-Octadecenoic acid, methyl ester, (Z)- (ODME)
8.	36.673	4.250	10,12-Hexadecadien-1-ol (HDO)
9.	36.816	8.380	6-Octadecenoic acid, (Z)- (OA)
10.	37.126	2.470	Octadecanoic acid (ODA)
11.	40.606	3.390	Tetradecanoic acid, dodecyl ester (TDDE)
12.	43.777	4.760	Hexadecanoic acid, tetradecyl ester (HATE)
13.	46.016	4.950	Hexadecadienoic acid, methyl ester (HAME)

Gravimetric measurements: Table-2 illustrates the inhibition efficiency of CSE (0.013 – 0.522 g/l) obtained by gravimetric measurements for Al in 1M HCl in 60 minutes exposure period at 35°C. The results showed that maximum inhibition efficiency of 82.4% was obtained at highest concentration (0.522 g/l) of inhibitor and trend of inhibition efficiency was linear with concentration of inhibitor. This behavior was due to increase in adsorbed inhibitor molecules over Al surface with the increment in inhibitor concentration leading to decrease in the oxidation of metal and hence corrosion. The inhibition efficiency (I %) and surface coverage (θ) was calculated using following equations:

$$I \% = \frac{W_u - W_i}{W_u} \times 100 \quad (1)$$

$$\theta = \frac{W_u - W_i}{W_u} \quad (2)$$

Where W_u and W_i is the weight loss of Aluminum in absence and presence of inhibitor.

According to previous research, the charge on metal surface is due to the oxidation of metal that emerges when it is dipped in the electrolyte solution. This surface charge could be positive or negative in nature. If the charge is negative then inhibitor molecules protonated by H^+ ions present in HCl solution will adsorb over metal. However, if the surface charge is positive then the inhibitor molecules through their electron rich sites will adsorb over the metal surface²². However, the rate determining step is the formation of a complex $[Al(OH)Cl]^+$ when corrosion of Al occurs in presence of HCl solution (electrolyte). To control the formation of complex, CSE was used as an inhibitor and molecules present in it inhibit the rate of corrosion. This

indicates that the electron rich heteroatom or π -electron present in inhibitor molecules might be getting adsorbed over the positively charged Al specimen.

Electrochemical impedance spectroscopy (EIS) measurements: EIS measurements was performed to study the inhibition behavior of CSE for Al in 1M HCl medium. Impedance result obtained in presence and absence of CSE as depicted in figure-1 was portrayed through Nyquist plot and table-3 provides the experimental data. The shape of the impedance curves was depressed semicircle. The result from the plot was evaluated using $R_s (QR_t [R_L L])$ as a circuit model. Where R_s , R_t , Q , R_L and L were solution, charge transfer resistance, constant phase element and inductive elements respectively. In EIS, efficiency of the inhibitor was obtained by considering R_t data of blank and inhibitor using following equation:

$$I \% = \frac{R_t - R_t^0}{R_t} \times 100 \quad (3)$$

Where, R_t and R_t^0 were the values of charge transfer resistance in existence of inhibitor in HCl solution and in blank solution respectively²³.

It can be observed from the result that the inhibition efficiency (only at lower concentration) value is higher in impedance measurements as compared to gravimetric one. The reason behind such a behavior may be the time difference during both the measurements which is 30 and 60 minutes in case of impedance and gravimetric calculations respectively.

Table-2
Corrosion parameters for pure Al in presence and absence of CSE at 35°C for exposure period of 60 minutes

Inhibitor	Inhibitor concentration (C) (g/l)	Weight loss mgcm ⁻²	Surface coverage (θ)	I %
Blank	-	23.685	-	-
CSE	0.013	21.273	0.102	10.182
	0.052	16.016	0.324	32.379
	0.104	13.089	0.447	44.736
	0.208	8.157	0.656	65.560
	0.261	7.967	0.664	66.361
	0.313	6.585	0.722	72.196
	0.365	5.420	0.771	77.116
	0.522	4.146	0.825	82.494

Table-3

Impedance parameters and corresponding inhibition efficiency for the corrosion inhibition of pure Al by CSE at 35°C

Inhibitor	Concentration (C) (g/l)	R_s ($\Omega \text{ cm}^2$)	R_t ($\Omega \text{ cm}^2$)	C_{dl} ($\mu\text{F cm}^{-2}$)	R_L ($\Omega \text{ cm}^2$)	L (Hcm ²)	n	I %
Blank		0.818	1.380	156.300	3.410	0.120	0.999	-
CSE	0.013	0.811	1.880	150.700	3.480	0.248	0.963	26.595
	0.052	0.890	2.262	150.200	4.190	0.554	0.967	38.992
	0.104	0.889	2.928	149.400	3.750	0.527	0.959	52.868
	0.208	0.861	4.070	147.000	7.220	0.379	0.930	66.093
	0.261	0.925	5.380	137.600	4.720	0.862	0.921	74.349
	0.313	0.888	5.390	134.000	11.820	0.633	0.932	74.397
	0.365	1.040	6.480	83.900	8.790	0.738	0.988	78.703
	0.522	1.065	7.420	83.790	7.580	1.367	0.983	81.401

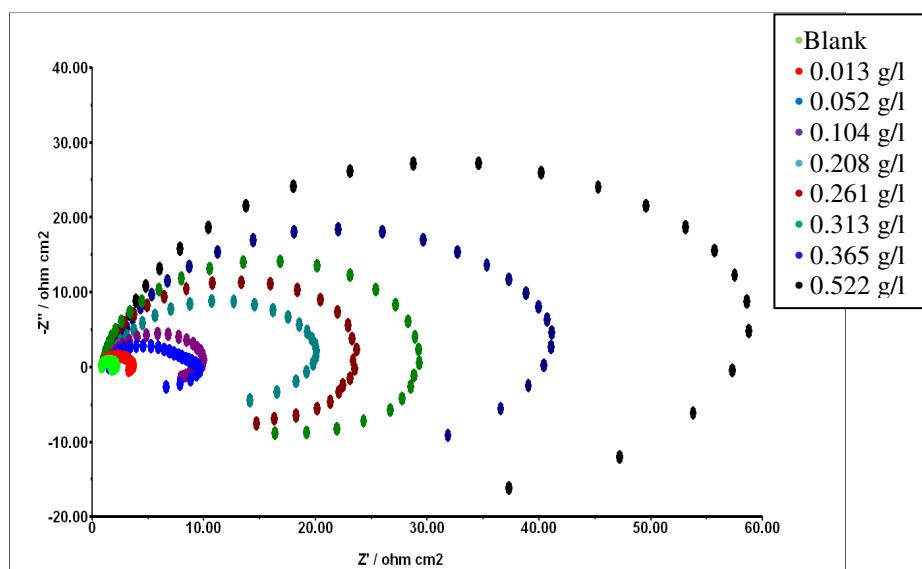


Figure-1

Impedance plot obtained at 35°C for corrosion inhibition of pure Al by CSE at various concentrations

The preceding research work proves that while studying EIS measurements for corrosion of Al in HCl medium, inductive loop can be observed at low frequency region. The reason behind inductive loop occurrence might be adsorption of some intermediates which causes reduction of hydrogen ion²⁴ or by formation of some inert film over Al surface²⁵. The presence of inductive loop was also observed in current work confirming its existence in Al corrosion when immersed in HCl solution.

The results reveal that R_t shows an increment in its value with inhibitor concentration. The reason behind such a behaviour was decrement in dissolution of metal ions (formed from its oxidation) into the electrolyte solution. When CSE was added in

the aggressive solution, these inhibitor molecules may come in between the metal and solution interaction region leading to decrease in metal ion dissolution and hence decrease in corrosion. However, Q was found to have inverse relationship with concentration of inhibitor. This clearly indicates that due to the adsorption of inhibitor molecules over metal surface, there was decrease in formation of electrical double layer and as the number of inhibitor molecules adsorbed were increased, larger area of metal surface will be covered by them leading to decrease in metal dissolution²⁶.

Adsorption isotherm: The study of adsorption isotherm was also performed in exemplifying the type of adsorption (physical

or chemical), CSE molecules followed for Al in 1M HCl medium solution at 35°C for exposure period of 60 minutes. Figure-2 depicts the Langmuir adsorption isotherm from which slope of 0.991 and regression coefficient of 0.997 was obtained indicating that CSE molecules follows Langmuir isotherm for inhibition of Al.

The equation of Langmuir isotherm is given below:

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \quad (4)$$

Where θ is degree of surface coverage, C (g/l) is concentration of inhibitor, K_{ads} is equilibrium constant for adsorption process²⁷.

The relationship of K_{ads} with standard free energy of adsorption (ΔG_{ads}^0) is depicted in equation 5:

$$K_{ads} = \frac{1}{C_{H_2O}} \exp\left(-\frac{\Delta G_{ads}^0}{RT}\right) \quad (5)$$

Where C_{H_2O} (g/l⁻¹), R (kJ mol⁻¹ K⁻¹) and T (K) are the concentration of water in solution, universal gas constant and temperature respectively. ΔG_{ads}^0 Obtained from the results was -23.16 KJ/mol. The stability and spontaneity of CSE molecules adsorption was confirmed from the data of free energy which was negative in nature. The calculated value of free energy is less than the threshold value (-40 KJ/mol) of chemisorption leading to the interpretation of physisorption mechanism.

Quantum chemical study: Inhibitor molecules can be theoretically investigated to understand their molecular reactivity using quantum study in framework of DFT²⁸. Figure-3 portrays the highest occupied (HOMO) and lowest unoccupied

(LUMO) molecular orbital of major CSE chemical constituents which provides information about the distribution of electrons in the respective compounds.

Figure-3 depicts that all the inhibitor molecules have surplus of electrons in the vicinity of π -electrons and O-atom except HDO where only π -bond has the electron donating capability. LUMO of the CSE molecules are located near π -bond in case of linalool, ODDA, ODME, HDO and OA while in case of ADD, TA, ODA, TDDE, TDA, HATE, HAME and HA, both π -bond and O-atom are capable of accepting electrons from filled orbital of metal.

DFT was used to calculate HOMO (E_{HOMO}) and LUMO (E_{LUMO}) energy of inhibitor molecules. Higher the E_{HOMO} value, greater is the inhibition property while lower values of E_{LUMO} show the same effect. In addition, energy gap (ΔE) indicates the energy required to remove electron from last occupied orbital. The lowest value of ΔE for a molecule indicates that the molecule had highest capability of showing inhibition. The energy values of molecular orbital of main chemical constituents of CSE are presented in table-4. It was observed that HDO has the highest value of E_{HOMO} and lowest ΔE . Therefore, HDO may contribute to corrosion inhibition efficiency larger as compared to molecules. In addition, fraction of the transferred electrons (ΔN) was also calculated and it was known that greater the ΔN value more will be the donating power of the molecule. According to previous study²⁹, if value of ΔN obtained was less than 3.6, the efficiency of inhibitor increases with E_{HOMO} . Therefore, it was observed from the result that HDO has highest ΔN value and E_{HOMO} indicating its maximum power to act as an inhibitor. Therefore, from DFT calculations the order of inhibitive activity of chemical compounds present in CSE can be: HDO>linalool>ODDA>OA>HAME>HATE>TDA>HA.

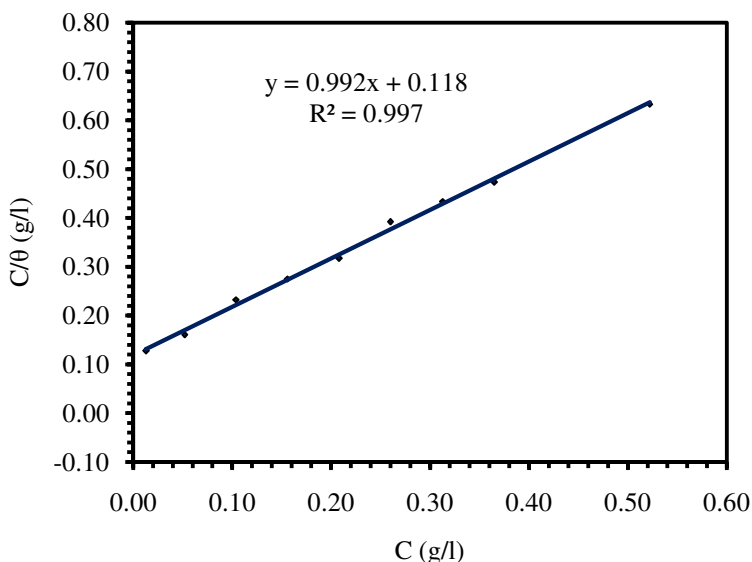


Figure-2
Langmuir adsorption isotherm plot for CSE at 35°C for 60 minutes

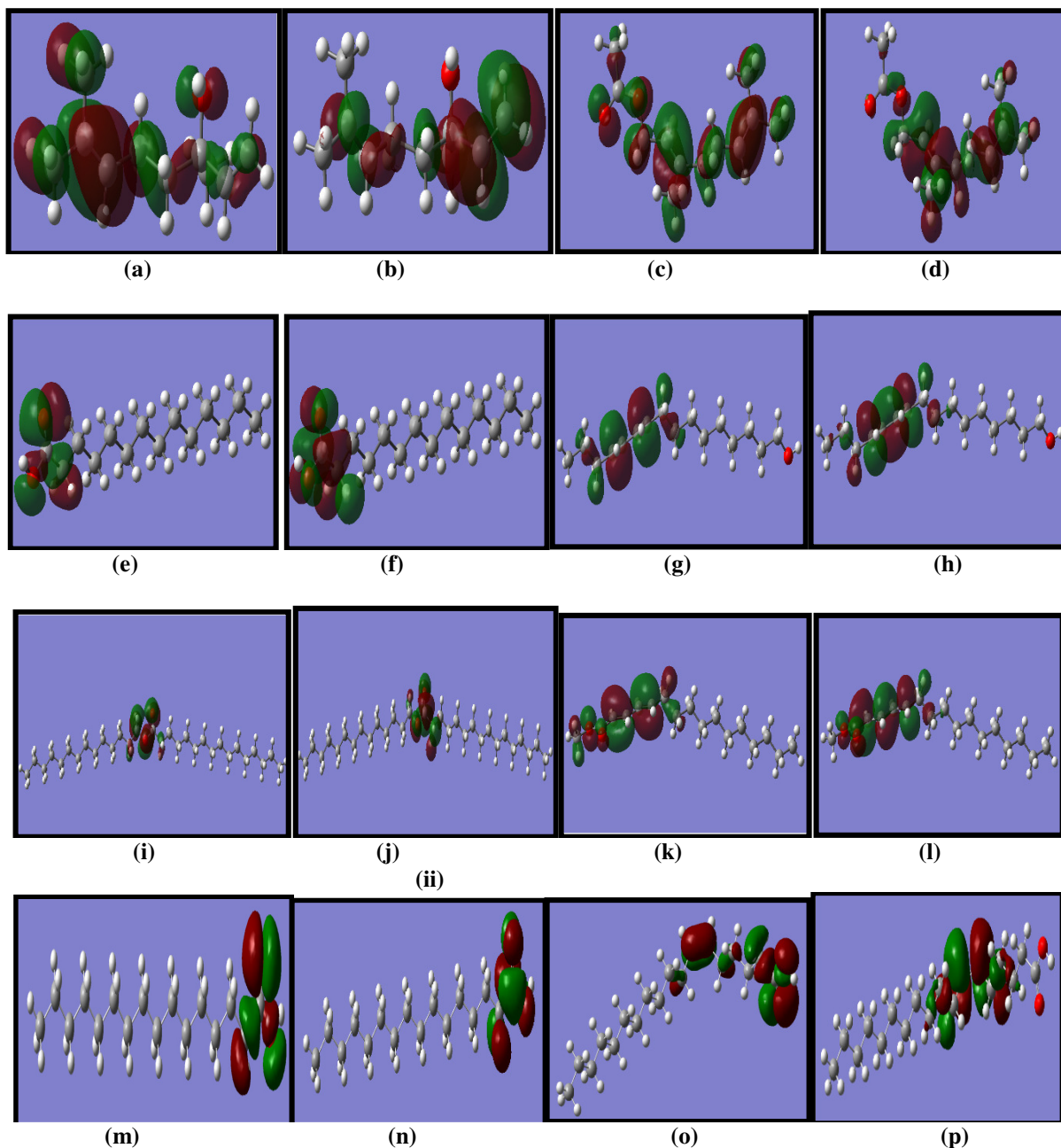


Figure-3

HOMO and LUMO of (a, b) linalool (c,d) ODDA; (e,f) TDA; (g,h) HDO; (i,j) HATE; (k,l) HAME; (m,n) HA; (o,p) OA

Conclusion

The inhibitive effects of various concentrations (0.013 – 0.522 g/l) of *CSE* were evaluated over pure Al in 1M HCl medium by using gravimetric and electrochemical impedance techniques. The result from gravimetric method showed an increment in inhibition efficiency with concentration of inhibitor. Langmuir adsorption isotherm was followed by *CSE* molecules and it was observed from its free energy value that the inhibitor molecules might be physisorbed over metal surface. The result from impedance measurements display that the constant phase

element decreases and charge transfer resistance value was raised with escalation in concentration of inhibitor. On basis of DFT calculation, HDO can be considered as the most probable inhibitive compound present in *CSE*.

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Table-4
Quantum chemical parameters of CSE

Compound	E _{HOMO} (eV)	E _{LUMO} (eV)	ΔE (eV)	ΔN (e)
linalool	-6.149	0.489	6.639	0.434
ODDA	-6.176	0.326	6.503	0.428
TDA	-7.428	0.326	7.754	0.279
HA	-7.428	-0.299	7.120	0.259
HDO	-5.605	-0.272	5.333	0.520
OA	-6.367	-0.217	6.149	0.394
HATE	-7.156	0.517	7.673	0.312
HAME	-6.285	1.524	4.761	0.379

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