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Synthesis and characterization of coumarin-3-amineas a green organic inhibitor complemented with good inhibitive performance

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Abstract : Green corrosion inhibitor as coumarin derived was synthesized by reaction of 2-hydroxybemzaldehyde and 2-acetamidoacetic acid. The targetcompound was characterized by elemental micro analysis (CHN), Fourier transform infrared spectroscopy (FT-IR) and protonNuclear magnetic resonance spectroscopy (NMR) spectra, elucidating that the eco-friendly corrosion inhibitor was effectively prepared. The corrosion inhibition efficiency of the green inhibitor in hydrochloric acid solution was estimated by weight loss technique.The inhibition efficiency was increased based on the increasing of the concentrations of the green inhibitor and become 86% with the highest experimental concentration, moreover the inhibition efficiency, decrease with the rising of temperature. Scanning electron microscopy (SEM) realization established that the inhibition of mild steel has been accomplished by adsorption of the eco-friendly inhibitor molecules on the steel surface and figuration of conservative film of the inhibitor on surface of the mild steel. Quantum chemical studies exhibit that the inhibitor has the tendency to be protonated in the corrosive solution and the results correspond with experimental noticing.
Keywords : N-(coumarin-3-yl)acetamide, coumarin-3-amine, Green corrosion inhibitor, eco-friendly.

Introduction

Mild steel is a material generally applied for the manufacture and transport in the gas and oil in industries because of the superior mechanical characteristics [1].Various problems appear during transportation of oil in the pipelines,where the ions migrating arrive to impinge with metal because of the denaturation of the emulsion solution (oil-aqueous), that induce corrosion approach [2] moreover, the corrosion is promote by the entity of salts in the oil and the acidic media that were utilized in exceedingly and oil well acidification [3]. In a higher corrosive medium, the corrosion approaches produce damage in the steel-structural. Various kinds of inhibitors that are vastly, utilized to hold the corrosion issue of mild steel at exposure to corrosive medium, that vary from organic molecules to nano-composites [4-7]. Various investigator have published that inhibition fundamentally relies on the electronic and physico-chemical characteristics of the inhibitor molecules, that are related to the entity of certain effective groups, steric effect and orbital characteristics for donate electron atoms. [7]. Metal surface charge also impact the adsorption behavior of molecules (inhibitors) [8]. Using inhibitors is one of the price-effective preservation techniques of mild steel in corrosive solutions. Commonly, the capability to produce strong coordination bond and, as a consequence, the inhibition efficiency increment based on the following way: Oxygen< Nitrogen < Sulfur < Phosphorous [9]. Survey of organic molecules that utilized as corrosion inhibitors are an important domain of investigation, due to its utility in different manufactures [10]. It has been published that organic compounds with heterocyclic rings have preferable and superior inhibition part in corrosive media [11]. Regarding to a continuation of prior investigations [12–16], we converge on the produce heterocyclic compounds as novel corrosion inhibitors. coumarin-3-aminehas been prepared in obviousness to be utilized as a corrosion inhibitor. The elucidation of the structure of coumarin-3-aminewas proved based on spectroscopical techniques named Fourier transform infrared spectroscopyandNuclear magnetic resonance spectroscopyand also elemental analysis. The using of coumarin-3-amineas corrosion inhibitor is based on the truth, of containing oxygen and nitrogen atoms would give more efficiently towards inhibition of corrosion of mild steel in corrosive solution. Structure for the coumarin-3-amineis demonstrated in Figure 1.

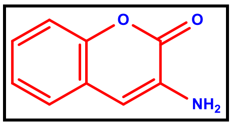

Figure 1. The structure of coumarin-3-amine.

Experimental

Chemistry:

All utilized materials in this work has been reagent grade that supplied by Sigma-Aldrich and/or Fluka and utilized without addition process. The Fourier transform infrared spectroscopy spectra had been carried out utilizing a FT-IR 8300 Shimadzu Spectrophotometer. Nuclear magnetic resonance spectroscopy spectra had been carried out utilizing on 300 MHz Bruker-DPX spectrometer with internal standard name TMS . Elemental micro analysis CHN, had been done using a 5500-Carlo Erba analyzer. A melting point apparatus model Gallenkamp M.F.B.600.010 F had been utilized to determine the melting point of corrosion inhibitor.

Synthesis of corrosion inhibitor (Coumarin-3-amine).

Asolution of acetic anhydride (0.5 mL, 0.053 mol) with β-acetamidoacetic acid (58.5 g, 0.5 mol), o-hydroxybenzaldehyde (92.7 g, 0.76 mol) and drops of piperidine had been refluxed for eight hours at 130 °C, then let to cooling at room temperature and separated out.Diethyl ether was utilized for washing, dried and using ethanol as recrystallization solvent to produce 85% yield ofN-(coumarin-3-yl)acetamide. An ethanolic solution of N-(coumarin-3-yl)acetamide (5 g, 0.02 mol) and concentrated HCl (2 mL) was refluxed for four hours. Product was cooled, poured on to ice and neutralization with sodium bicarbonate then filtered. Ethanol was utilized as recrystallization solvent to give a pale yellow powder,yield 17%, m.p. 129 °C; $NMR_{spectroscopy}$: σ 6.11 (s, for 1H of alkene); σ 7.40-7.61 (m,for 1H ofbenzene ring); σ7.97 (s, amine).$IR_{spectroscopy}$: 3407$cm^{-1}$with 3299 cm^{-1} for amino group and 1709 cm^{-1} for carbonyl group.$CHN_{analysis}$ for $C_9H_7NO_2$ were C 66.98% (C 67.07%) H4.27% (H 4.38%), N 8.58% (N 8.69%).

Weight loss measurements:

The acquired specimens of Mild steel were from Company named Metal Samples had been utilized as working electrodes investigation and they were with composition (wt%) as follows: Fe, 99.21; C, 0.21; Si, 0.38; P, 0.09; S, 0.05; Mn, 0.05; and Al, 0.01. Specimens had been cleaned based on to ASTM standard methodology G1-03 [30]. All experiments were cried out in aerated, non-stirred one normal o hydrochloric acid solutions with various concentrations of coumarin-3-amineas an inhibitor.The mild steel specimens of 25 mm × 20 mm × 0.25 mm were washed with double distilled water, rinsed with ethanol and acetone, and then dried at room temperature. After weighing accurately, thespecimens were suspendedin1000 ml of 1.0 M HCl solution with various concentrations of the coumarin-3-amine (0.0, 0.05, 0.1, 0.15, 0.20, 0.25 and 0.50 mM) for 1, 2, 3, 4, 5, 10, 24 h. After each immersion time, the specimens were taken out, washed, dried, and weighed accurately. The weight loss measurements were carried out in triplicate.

Scanning Electron Microscopy Morphologies:

A scanning electron microscope screening was performed at the Electron Microscopy Unit/UKM utilizing SEM/TM1000 Hitachi Tabletop Microscope at 2000× magnification. Investigation of the mild steel samples was done for immersed in HCl, in presence and absence of coumarin-3-aminefor three hrs. Scanning Electron Microscopy (SEM). The morphology of the surface of the metal samples had been investigated via SEM immediately after the weight loss techniques.

Results and discussion

The preparation of coumarin-3-amine was done by refluxing vigorously of the mixture ofo-hydroxybenzaldehyde and β-acetamidoacetic acid, with strong base named piperidine. The reaction would end through of acetyl group removal and formation of coumarin-3-aminewith 17% yield. The sequence for this reaction that represent the reparation of ec-friendly inhibitor derived from coumarin has been outline in Figure 2).

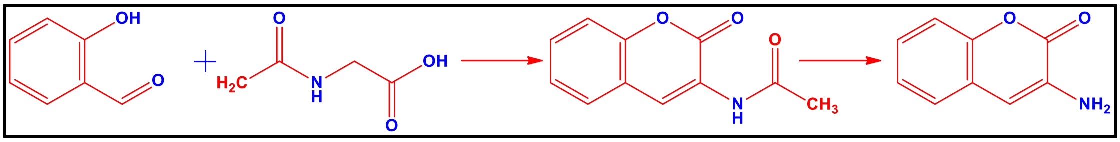

Figure 2. Preparation of coumarin-3-amine.

The FT-IR spectrum of coumarin-3-amine demonstrated absorption bands for amino group at 3407cm-1 and 3299 cm-1. The 1H-NMR spectrum exhibited a singlet at δ 6.11 ppm because of one proton for alkene group. Reaction mechanism could be regarding to a carbanion mechanism as in Figure 3.

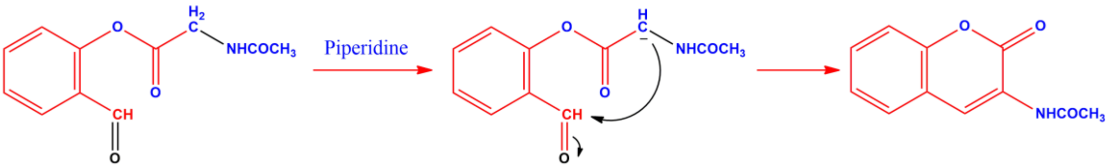

Figure 3. The reaction mechanism or the formation of coumarin-3-amine

Concentration Impact

The impact of increment of coumarin-3-amine in corrosive solution on the corrosion of mild steel was examined based on weight loss technique for a period of time (1, 2, 3, 4, 5, 10 and 24 h) at 303 K. The values of corrosion rate and inhibition efficiency with and without coumarin-3-amine were shown in Figures 4 and 5. The corrosion rate and The inhibition efficiency IE (%) had been estimated based on Equations 1 and 2 respectively:

$$C_R(Corrosion\ rate) = 87.6\ W/at\rho \qquad (\mathbf{1})$$

where w is the weight loss, ρ is the density of mild steel, a is the area of specimen and t is the time of immersion.

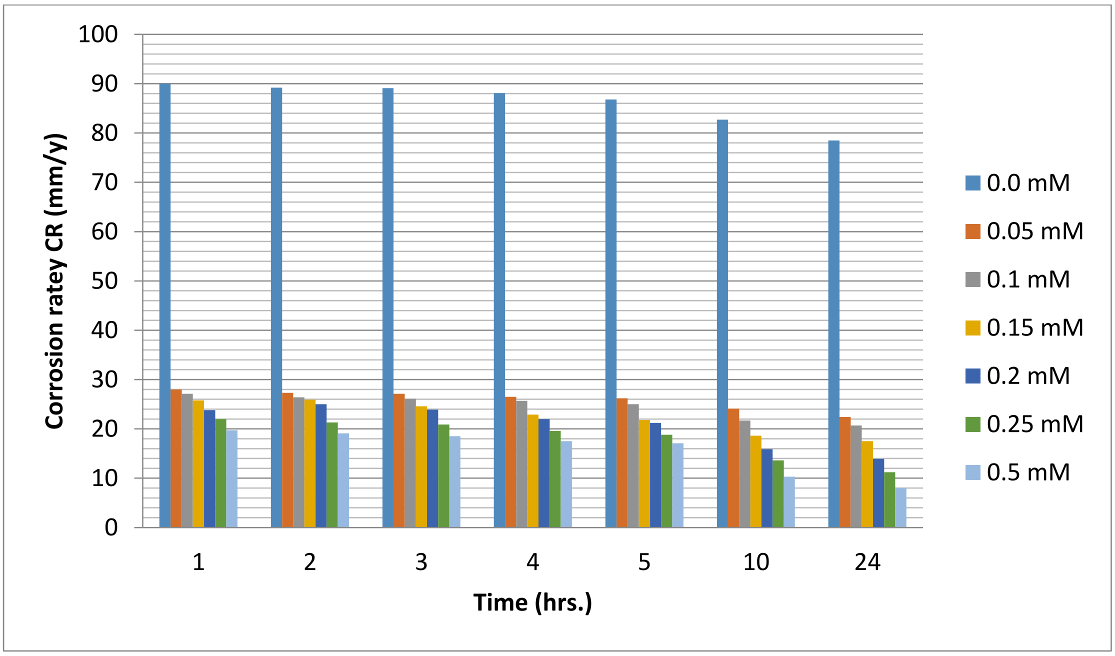

Figure 4. Impact of coumarin-3-amine concentration andtime on corrosion rate of mild steel at 303 K.

$$\text{IE\% (Inhibition Efficiency)} = \frac{w_1 - w_2}{w_1} \times 100 \qquad (\mathbf{2})$$

where the W and W' were the weight of the mild steel samples without and with inhibition.

The corrosion rates were markedly decreased and the inhibition efficiency was enhanced with the increasing concentration of coumarin-3-amine. The improvement of IE(%) withthe higher concentration is revealing of the raise in the range of protection efficiencies of coumarin-3-amine.

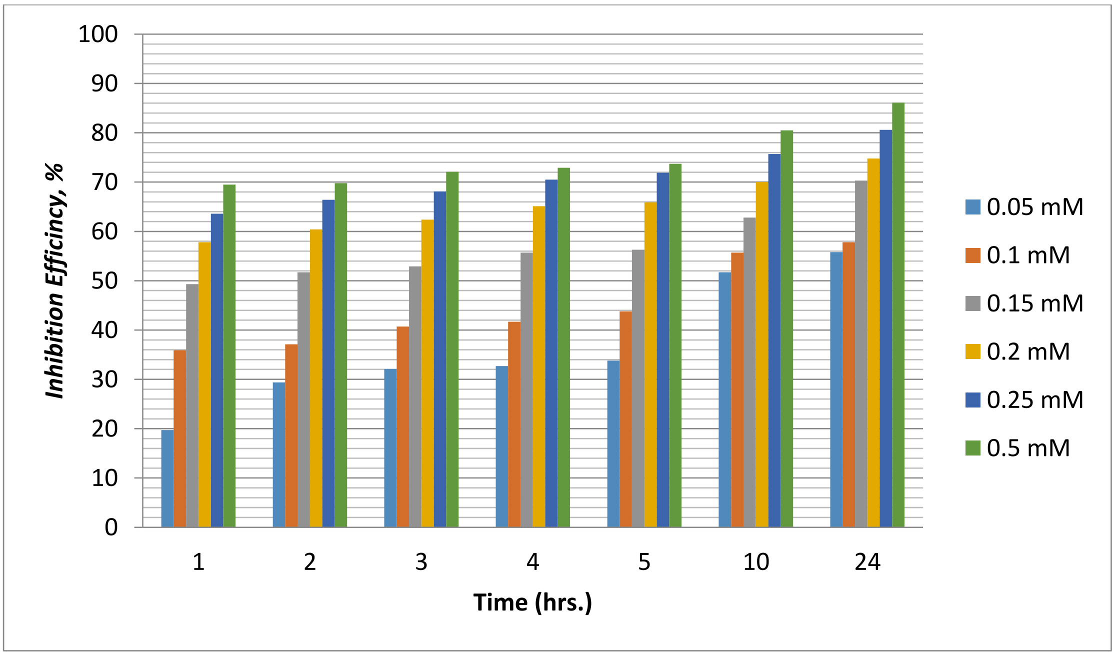

Figure 5. Impact of coumarin-3-amine concentration and time on inhibition efficiency of mild steel at 303 K.

Temperature Impact

A differentiation of the inhibition efficiency of coumarin-3-amine on mild steel in corrosive solution with various concentrations (0.0, 0.05, 0.1, 0.15, 0.20, 0.25 and 0.50 mM) of coumarin-3-amine at temperatures (303, 313, 323 and 333 K) indicated that IE improved with enhancement of concentration of coumarin-3-amine and also decreased with higher temperature as shown in Figure 6.For the adsorption process of inhibitor molecules, the heat of adsorption is markedly negative, and this specified an exothermic reaction. This is the cause that the inhibition efficiency decreases at a higher temperature.

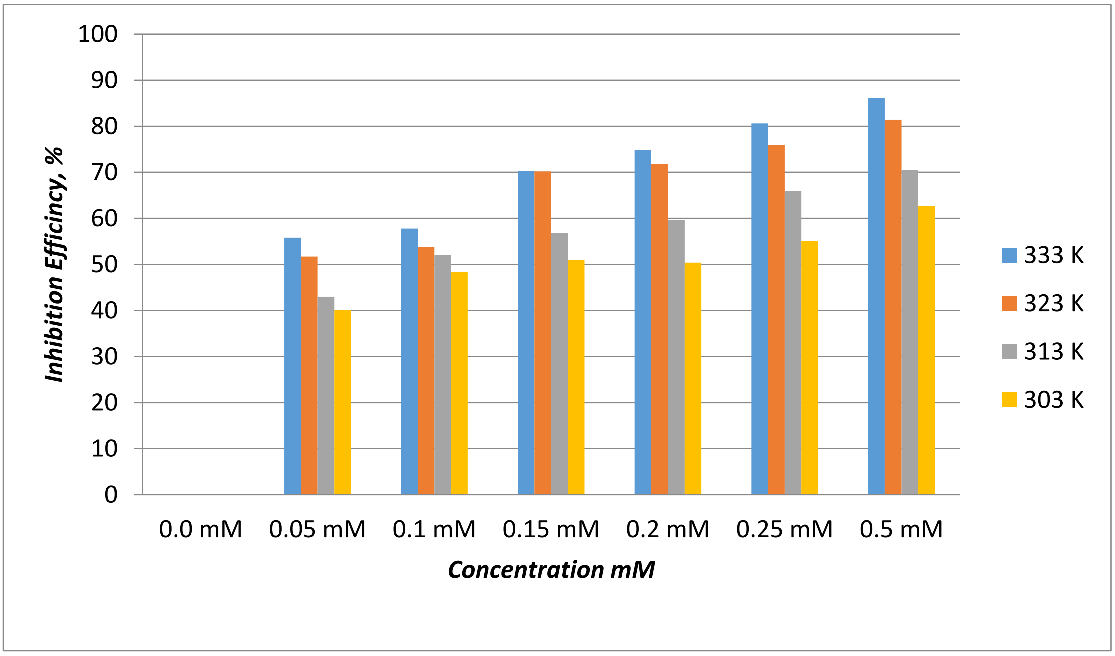

Figure 6. Impact of temperature on inhibition efficiency of coumarin-3-amine at various concentrations

SEM

Regarding to Figure7, as predictable, significant corrosion of mild steel appeared where the MS (mild steel) surface, that was primarily smooth and clean, turn into harsh. The MS surface was markedly attacked by corrosive solution. Regarding to Figure 8, MS surface dose not afford,significant corrosion. Coumarin-3-amine has the ability to completely protective potentiality the MS from the corrosion exposure relevant by corrosive solution.

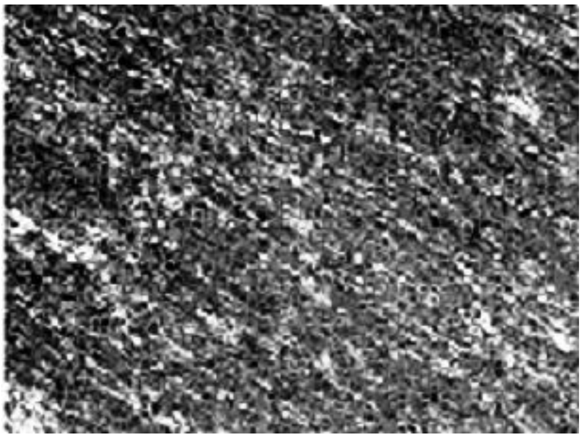

Figure 7. The SEM micrograph for mild steel in 1.0 M HCl at 30 °C for 5 h as immersion time.

Figure 8. The SEM micrograph for mild steel in 1.0 M HCl with 0.5 mM of coumarin-3-amine at 30 °C for 5 h as immersion time.

Postulated mechanism

Coumarin-3-amine is adsorbed on the MS surface to produce a thin film as a completely protective potentiality and/or coordination bonds through the reaction of coumarin-3-amine as an inhibitor and MS as a metal. The adsorption mechanism of coumarin-3-amine could conduct through one of three channels. The first channel, is the charge of coumarin-3-amine molecules and electrostatically attract o the metal. Second channel, represented by the interaction of unshared electrons of oxygen and/or nitrogen atoms with the metal.Third Channel is the interactionof π-electrons of double bond of coumarin-3-amine and the metal surface. Coumarin-3-amine may protect the metal surface via blockingcathodic and/or anodic toproducemetal complex. Inhibition efficiencies of coumarin-3-amine of the corrosion of mild steel in corrosive solutionmay be demonstratedbased on the adsorption sites, charge, size of molecules, interaction withmetal and ability of metal complex. The π electrons for the double bond and free electrons on the oxygen and nitrogen atoms form chemical bonds with the metal surface as shown in Figure 9.

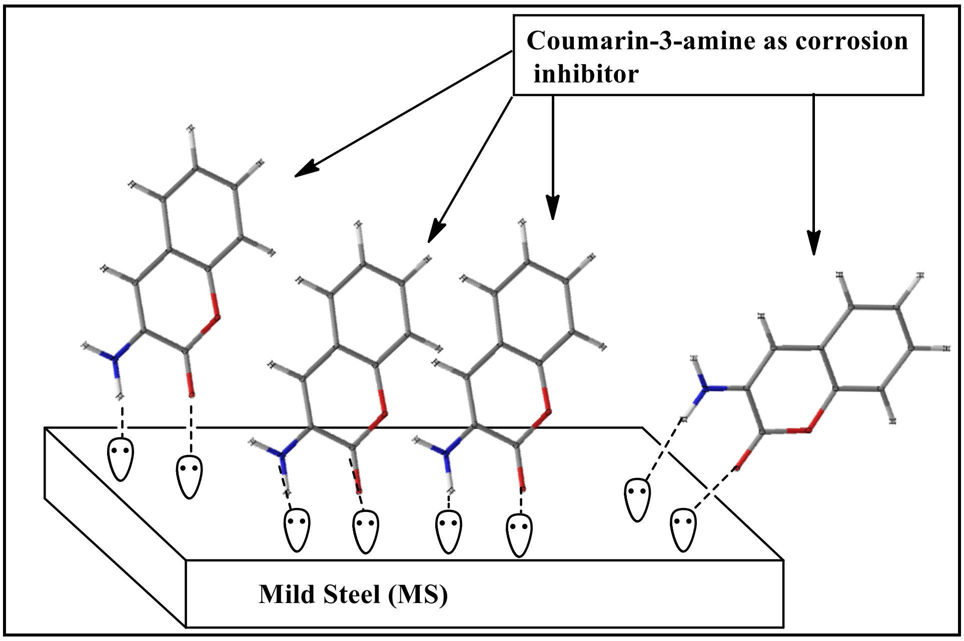

Figure 9. The postulated inhibition mechanism for coumarin-3-amine

Adsorption Isotherm

Generally adsorption rely, on the nature of the metal surface, electronic properties of the metal, adsorption of solvents and ionic species, on the electrochemical potential at solution interface. The adsorption mechanism of inhibitors molecules on MS surface could be demonstrate based on the investigation of adsorption isotherm and behavior of the inhibitor. The generality frequently utilized adsorption isotherms are the Langmuir, Temkin, Frumkin, and Freundluich isotherms [31]. Corrosion inhibition of inhibitors on MS corrosive solution could be explained according to a molecular adsorption technique. The adsorption method is affected by the molecular structure of inhibitor with distribution of charge on molecules in addition to nature of the MS surface and the corrosive media [32].The significance of surface coverage (θ) for the various concentrations of coumarin-3-amine had been utilized to show the worth adsorption isotherm to determine the adsorption process. To estimate θ, it was supposed [33] that the IE (%) was appropriate generally to the blocking impact of the adsorbed species as in Equation (3) applies:

$$\theta = IE\%/100 \qquad (3)$$

In this investigation, θ was estimated from the equation 3, utilizing the IE that estimated from the weight loss method. The plots of C_{inh}/θvs C_{inh}yield a straight line, referencing that coumarin-3-amine obeys the Langmuir adsorption isotherm, as inthe Equation (4):

$$C_{inh}/\theta = 1/k_{ads} + C_{inh} \qquad (4)$$

where $C_{inh}$is coumarin-3-amine concentration and $k_{ads}$is the adsorption constant gained from the intercept of the straight line.

k_{ads} is correlating with the standard free energy of adsorption ΔG^o_{ads}.

$\Delta G^o_{ads}$is assumed by means of Equation (5):

$$\Delta G^o_{ads} = -RT ln[k_{ads}] \qquad (5)$$

where the value of 55.5 demonstrate the molar concentration of water in solution obvious in units of M. R is the universal gas constant and T is the absolute temperature.

The value of *Kads* and ΔG^o_{ads} ware estimated based on Figure 10 and the estimated $\Delta G^o_{ads}$was−24.75 kJ/mol. The negative[34-38]charge of ΔG^o_{ads}indicate spontaneously adsorption of the coumarin-3-amine on MS surface and a strong interactionsof coumarin-3-amine molecules and the MS surface. Usually, a value of ΔG^o_{ads} nearly −20 kJ/mol is consistent with physical adsorption, but value of ΔG^o_{ads}nearly −40 kJ/mol is chemical adsorption occurring viatransfer of unpaired electrons from coumarin-3-amine to MS.

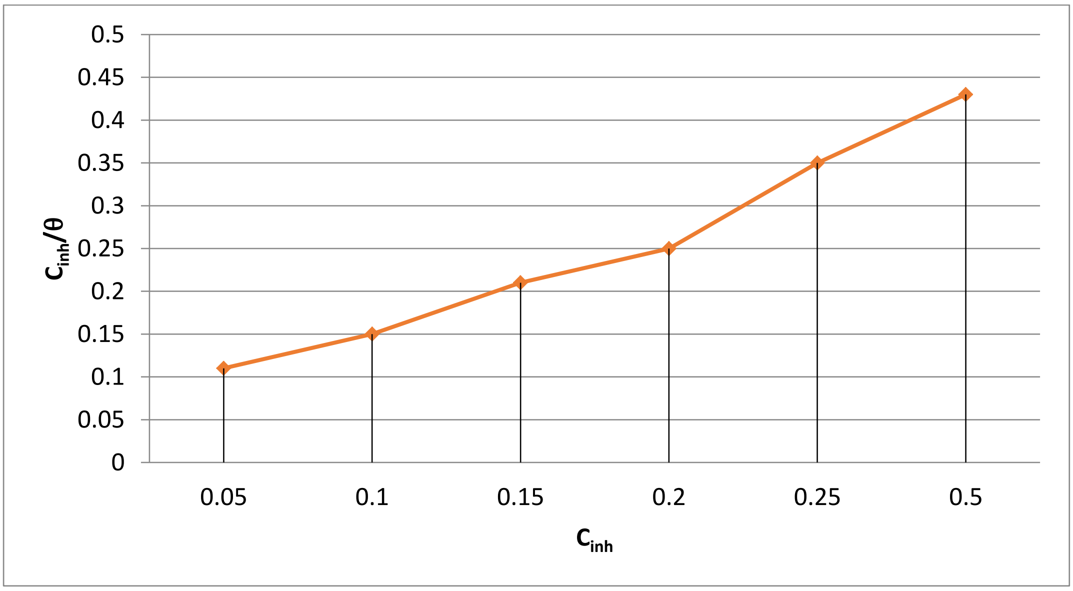

Figure 10. Adsorption isotherm for mild steel in 1.0 M HCl with different concentrations of the corrosion inhibitor.

Conclusions

The investigation results implied that coumarin-3-amine was a perfect corrosion inhibitor for mild steel in corrosive solution in a conditional concentration. Inhibition efficiency of coumarin-3-amine at the maximum concentration was up to 86.1%reducing with a higher temperature degrees. Coumarin-3-amine is adsorbed by mild steel surface that obey Langmuir/isotherm. Coumarin-3-amine is demonstrate as an effective inhibitor having perfect, inhibitive characterstics becuse of oxygen and nitrogen atoms. SEM analysis confirmed the forming of a barrier layer from coumarin-3-amine on the mild steel surface. The anti corrosion investigation of coumarin-3-amine obviously demonstrated its controlling protection of MS in corrosive solution.

Competing Interests: The authors have declared that no competing interests exist.

Acknowledgments

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Author Contributions

Conceived and designed the experiments: AAA . and WKA; Performed the experiments: WKA and AQS. Analyzed the data: AAA. Contributed reagents/materials/analysis tools: AAA and KSR. Wrote the paper: AAA and KSR.

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