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Experimental and Theoretical Studies of One Dihydropyridine Derivative as Corrosion Inhibitor in Acidic Media

Homa Shafiekhani[1*], Somayeh Karimi[2], Mohammad Torkashvand[3]
1. Department of Chemistry, Payam Noor University, P.O. Box 19395-4697, Tehran, Iran
2. Islamic Azad University, Lamerd Branch, P.O. Box 74341-553881, Lamerd, Iran
3. College of Engineering, University of Tehran, P.O. Box. 14395-515, Tehran, Iran
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Abstract
A new organic compound, namely dihydropyrimido [4,5-b][1,6] naphthyridine-2,4, 6, 8(1H,3H,7H,9H)-tetraones with amino acid moiety (DHPN) was synthesized and characterized by ^{1}H, ^{13}C Nuclear magnetic resonance (NMR) and Fourier transform infrared (FTIR) spectroscopy experiments. DHPN was investigated for the first time as a green inhibitor of mild steel (A105) corrosion in acidic (0.1, 0.5 mol L^{-1} H_2SO_4 and HCl) solutions using potentiodynamic polarization technique. The results showed that, inhibition efficiency increased with the inhibitor concentration within the range of 0.95-19 mg L^{-1}. The polarization curves demonstrated that, this compound act as a mixed type inhibitor. The adsorption of the DHPN molecule on the surface of mild steel was found to obey the Langmuir adsorption isotherm. Besides, data processing methods like support vector machine modelling was performed to prove the relationship between inhibitory effect and molecular structure.

Keywords
Corrosion Inhibition; Support Vector Machine; Mild Steel; Potentiometric Polarization.

1. INTRODUCTION
One of the most important manufacturers of pipelines in the gas and oil industry is carbon steel. Corrosion of these pipelines in acidic environments is a serious problem. Destructive solutions such as hydrochloric and sulphuric acids, which are used for acid pickling leads to corrosive attack [1, 2]. Using inhibitors is one of the best techniques to prevent corrosion of metals. The addition of these compounds in low concentrations can reduce or prevent corrosion. Although many inhibitor compounds have prevented corrosion but organic inhibitors are less toxic than inorganic compounds. Organic compounds containing heteroatoms like (O, S, P, and N) are efficient against metallic corrosion in wet corrosion environments [3-8]. Organic inhibitors can be adsorbed onto the metal surface through chemical interaction and physical adsorption and reduce corrosion rate [9-13]. These compounds can form covalent bonds or electrostatic interactions between the metal surface and the inhibitor [14]. Then, it is necessary to find an alternative to them with minimum toxicity and environmental friendly [15]. Nowadays, most researchers are trying to find environmentally friendly inhibitors. L-amino acids, due to the characteristics of high safety, biodegradable, relatively cheap and soluble in aqueous media, as a green corrosion inhibitor have been noticed [16-17]. In particular, cysteine ($HSCH_2CHNH_2COOH$) stands as one of the most promising candidates because this molecule contains the thiol group [-SH], the amino group [-NH_2] and, the carboxyl group [-COOH].

In this work, we synthesized a new organic compound, namely dihydropyrimido [4, 5-b] [1, 6] naphthyridine-2, 4, 6, 8(1H, 3H, 7H, 9H)-tetraones with amino acid moiety (DHPN) for the first time as a corrosion inhibitor for mild steel in acidic media (HCl and H_2SO_4). Corrosion inhibition efficiency of carbon steel in (0.1, 0.5M H_2SO_4 and HCl) acid solutions was studied by using potentiodynamic polarization. Besides, we used support vector machines (SVM) to evaluate inhibitor efficiencies obtained from the experimental studies [18].

2. EXPERIMENTAL
2.1. Material and Methods
The steel sample utilized as a part of this study (A105) was cut from petroleum pipelines, with a chemical composition (in wt.%) of 0.35% C, 0.6-1.05% Mn, 0.035% P, 0.04% S, 0.1-0.35% Si, 0.3% Cr, 0.08% V, 0.4% Ni, 0.009 % Mo, 0.4% Cu and balance Fe.

The test solutions (0.1, 0.5 mol L^{-1} HCl and H_2SO_4) were prepared by dilution of concentrated HCl (37% wt.) and H_2SO_4 (98% wt.) with double distilled water, respectively. The concentration range of inhibitors employed was varied from (0.95 to 19 mg L^{-1}). The ^{1}H-NMR spectra were

*Corresponding Author: shafieehoma@yahoo.com

recorded in $CDCl_3$ solvent with a Bruker DRX-500 spectrometer and FT-IR spectroscopy was performed with a Jasco 480 plus spectrometer.

The electrochemical measurements were done using an µAutolab III (PGSTAT30) potentiostat/ galvanostat and controlled by NOVA1.5 programming. The electrochemical cell included a three-electrode setup where a platinum electrode was used as a counter electrode, the reference electrode was a saturated calomel electrode (SCE), and the working electrode (steel sample) was carbon steel (A105), with the surface area of the electrode 1.00 cm^2.

2.2. Synthesis of dihydropyrimido [4, 5-b][1, 6] naphthyridine-2, 4, 6, 8(1H, 3H, 7H, 9H)-tetra ones (DHPN)

In a 5 mL round bottom flask equipped with a condenser, 3-Hydroxy-4-methoxy benzaldehyde (1 mmol), barbituric acid (2 mmol), 12-tungstophosphoric acid (TPA) as the catalyst (0.1 mol), and ethanol as the solvent (3 mL) were mixed. The mixture was refluxed at 80° C for 12 hours. Then L-cysteine (1 mmol) was added and the mixture was refluxed overnight. The precipitated product was filtered, and washed three times with 2 mL ethanol. The molecular structure of DHPN is shown in Fig.1.

IR (KBr): ν/cm^{-1}: 3258, 3247, 3089, 1728, 1671, 1410, 1356, 1323, 1275.

^{1}HNMR (DMSO-d6) δ_H (ppm): 2.2 (2H, S), 2.7 (3H, S), 3.14 (2H, d), 5.53 (1H, t), (6.1-6.5) (3H, m), 8.2 (1H, S), 9.7 (4H, S), 16.95 (1H, S).

^{13}CNMR (DMSO-d6) δ (ppm) =8.5, 29.5, 29.6, 45.6, 55.6, 91.1, 111.7, 114.4, 116.9, 117, 137.4, 144.6, 145.6, 150.6, 162.4, 163.7.

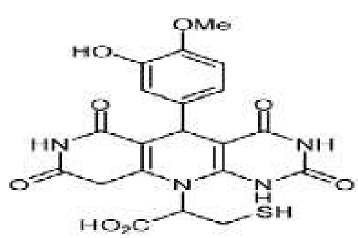

Fig. 1. Structures of DHPN Formula Weight=475

2.3. Electrochemical experiments

Before different experiments, the sample surface was polished with emery papers, washed, and degreased in acetone, dried and weighed. Before measurements of polarization curves, the working electrode was immersed in the test solution with and without inhibitor for 30 minutes to set up a steady-state open circuit potential (E_{ocp}). The polarization curves were recorded in the potential range from -250 to 250 mV at the scan rate of 1 mVs^{-1}. All potentials were recorded with concern to the SCE. All experiments were done at temperature 298°K.

3. RESULT AND DISCUSSION

3.1. Electrochemical measurements

To investigate the effect of the inhibitors on carbon steel corrosion, potentiodynamic polarization (PDP) studies were performed. Fig. 2 showed the Tafel polarization curve for carbon steel in an acidic medium (0.5 mol L^{-1} H_2SO_4) solution containing the different concentration of DHPN (0.95-19 mg L^{-1}).

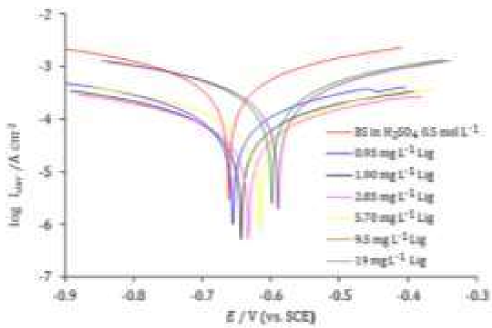

Fig. 2. Typical polarization curves for sample steel in (0.5 mol L^{-1}) H_2SO_4 for various concentrations of DHPN at 298° K and scan rate, 1 $mVsec^{-1}$.

The electrochemical parameters such as corrosion potential (E_{corr}), current density (I_{corr}), Tafel slopes cathodic and the anodic (β_c and β_a), and inhibition efficiency (IE, %) are given in Table 1. The surface coverage degree (θ) and efficiency of inhibition (IE, %) were obtained according to Equations. (1) and (2) respectively [19-21]:

$$IE\% = \left(\frac{I^0_{corr}-I_{corr}}{I^0_{corr}}\right) \times 100 \quad (1)$$

$$\theta = \frac{IE\%}{100} = 1 - \frac{i_{corr}}{i^0_{corr}} \quad (2)$$

Where I_{corr} and $I°_{corr}$ are the corrosion current densities with and without the inhibitors, respectively. As can be seen, after the addition of inhibitor (DHPN), anodic and cathodic current shift to lower current densities. That indicated the presence of inhibitor reduced anodic dissolution of carbon steel and also retarded the cathodic reaction [22-23]. Inhibitors can be classified as anodic, cathodic or mixed type according to E_{corr} values. In literature, it has been reported that if the corrosion potential (E_{corr}) value of the inhibited solution is more than ±85 mV with concern to the blank solution, the inhibitor can act as an anodic or cathodic inhibitor [24, 25]. In this study, the maximum displacement in E_{corr} is 62 mv in the presence of DHPN. Thus, it is concluded that the DHPN molecule behaves as mixed-type inhibitor with mostly anodic inhibitive action.

Table 1. The corrosion parameters obtained from polarization plots carbon steel (A105) in 0.5 mol L^{-1} H_2SO_4 containing different concentrations of DHPN

C (mgL^{-1})	E_{corr} (V)	I_{corr} (mA cm^{-2})	B_c (V dec^{-1})	B_a (Vdec^{-1})	CR (mpy)	θ	IE (%)
0	-0.661	0.710	0.498	0.485	325.37	----	----
0.95	-0.655	0.166	0.583	0.470	76.04	0.766	76.63
1.90	-0.643	0.096	0.464	0.413	44.22	0.864	86.41
2.85	-0.632	0.080	0.445	0.408	36.52	0.888	88.80
5.70	-0.616	0.094	0.382	0.344	43.04	0.868	86.77
9.50	-0.599	0.201	0.214	0.259	92.13	0.717	71.70
19.0	-0.589	0.210	0.226	0.292	96.40	0.704	70.40

Furthermore, when the inhibitor concentration increased, the corrosion current density of carbon steel reduced from 0.710 mA cm^{-2} to 0.080 mA cm^{-2}, and the corrosion inhibition efficiency increased to 88.80%. This behavior is the excellent coverage of the metal surface by DHPN molecule, which blocked the reaction sites on the metal surface. The anti-corrosion properties of organic inhibitors for mild steel in acidic media can be explained based on the molecular structures of the studied inhibitors. Indeed, it depend on many factors, including their concentration, the number of active sites and their corresponding charges, molecular mass and, their stability in corrosive environments. Furthermore, the presence of heteroatoms with lone pairs of electrons (N, O and S) in this compound could donate their free electron to the unfilled steel atom orbitals, and the inhibitor adsorbed on the mild steel surface.

3.2. Adsorption isotherm

The adsorption isotherm gives information about the interaction between inhibitor, and the carbon steel surface. Several adsorption models (Langmuir, Temkin and Freundlich) were examined for to interpret of corrosion inhibition [26]. We noticed that the adsorption of DHPN on the metal surface in sulphuric and hydrochloric acid solutions obeyed the Langmuir adsorption isotherm [27]. This was represented by Equation. (3) [27]

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \qquad (3)$$

Where C = inhibitor concentration (mg L^{-1}), θ = degree of surface coverage on the metal surface and K_{ads} = equilibrium constant for adsorption-desorption process. The linear variation of C/θ vs. C_{inh} of the ligand DHPN in 0.5 mol L^{-1} H_2SO_4 solution showed in Fig. 3. The good correlation coefficient and the linearity that appear at the plot suggest that the plot obeys the Langmuir adsorption isotherm [28]. The R^2 value is very close to unity, indicating a strong agreement with the Langmuir adsorption isotherm. The other plots related to Temkin and Freundlich isotherms were showed in supplementary files.

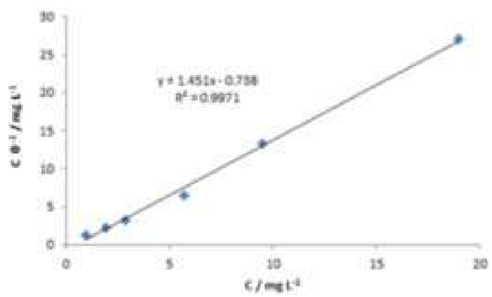

Fig. 3. Langmuir isotherm for the adsorption of inhibitors on the carbon steel surface in (0.5 mol L^{-1}) H_2SO_4 at 298°K.

3.3. Performance evolution

To evaluate the performance and accuracy of the new proposed correlations and previously discussed ones, both statistical and graphical error analysis have been utilized simultaneously.

3.4. Statistical parameters analysis

To assess the accuracy and performance of the existing correlations and newly developed ones, some statistical parameters defined including, average percent relative error, average percent absolute relative error, the standard deviation of error, mean square error and coefficient of determination.

3.5. Modelling of data using support vector machine (SVM)

Measuring and testing independence are necessary in mathematical modelling, statistical modelling and experimental sciences [29]. In data processing tools, the independence parameter is assigned a project as a normal variable.

SVM is a classification method in which the training datasets are separated by a hyperplane with a maximum distance from the assist vectors. The method of the Lagrangian multiplier is employed to a maximise the margin. A complicated set of data, different kernel functions consisting of linear, radial basis function, sigmoid and polynomial are employed to map the information into an alternative higher dimensional space [30]. An appropriate kernel function can have a widespread impact on the capacity of the SVM [31]. In contrast to different classes of neural networks, this technique can undergo over fitting and under fitting [32]. As we noted earlier, we used the SVM model for modelling the Cysteine data and, so the consequences were received in accordance with this model as we confirmed in Fig. 4-6.

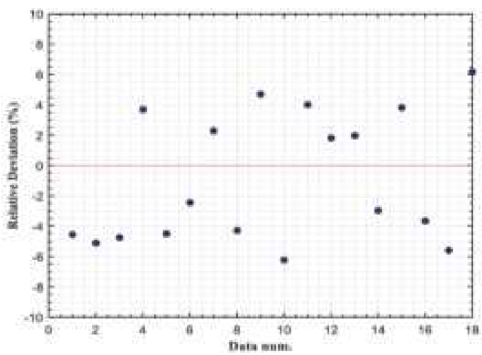

Fig. 4. Comparison of relative deviation of artificial intelligence and Experimental data of IE %

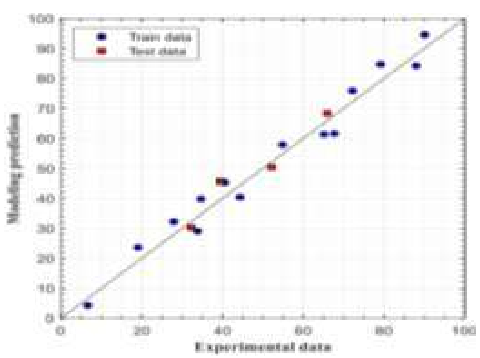

Fig. 5. A comparison between experimental data and calculated ones from SVM model

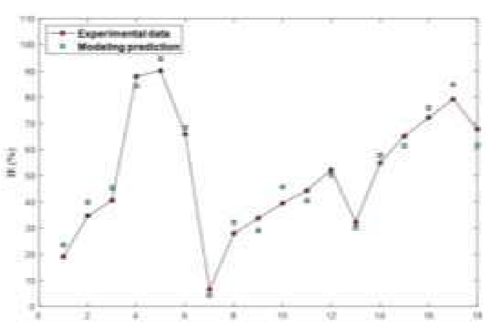

Fig. 6. Experimental data vs. predicted data of obtained IE % with SVM model in training and validation

4. CONCLUSION

The organic inhibitor namely, DHPN was tested for its corrosion inhibition performances on (A105) pipelines steel in (0.1, 0.5) M H_2SO_4 and HCl solutions using electrochemical techniques. From the results of the study, DHPN showed excellent corrosion inhibition performances for mild steel (A105) in acidic media. The inhibition efficiency increased with increasing concentration of the inhibitor and reached a maximum of 88.8% in the presence of 2.85 mg L^{-1} of inhibitor. The corrosion rate significantly decreased and adsorption behavior of DHPN on carbon steel surface in acidic media obeyed Langmuir adsorption isotherm. The inhibition mechanism of occurs through adsorption processes DHPN on the metal surface to form a thin protective layer. Potentiodynamic polarization reveals that inhibitor retards both anodic and cathodic reactions on the surface of the metal. Thus, polarization measurement suggests that the DHPN is a mixed type inhibitor. The obtained results by neural network technique for modelling the experimental data using the SVM method confirm experimental results.

The SVM modelling results of test data (Figures 4 to 6), which have been compared to train & test data for convenience, show a close approximation, i.e., very low error in these numbers. This case (low number difference and low yield error) confirms the claim that SVM modelling has been able to pursue its goal correctly.

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مطالعات آزمایشگاهی و تئوری مشتقی از خانواده دی هیدرو پیریدین به عنوان بازدارنده خوردگی در محیط های اسیدی

هما شفیع خانی[۱و*]، سمیه کریمی[۲]، محمد ترکاشوند[۳]
۱. گروه شیمی، دانشگاه پیام نور، تهران، ایران
۲. دانشگاه آزاد اسلامی واحد لامرد، لامرد، ایران
۳. دانشکده مهندسی، دانشگاه تهران، تهران ، ایران
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چکیده
یک ترکیب آلی جدید از خانواده بی پیریدین‌ها با استخلاف آمینو اسید (گروه عاملی سیستئین) سنتز شد و ویژگی آن با طیف‌های ان ام آر و آی آر تایید شد. این لیگاند سنتزی برای اولین بار به عنوان یک بازدارنده خوردگی لوله‌های استیل در محیط‌های اسیدی (اسید سولفوریک و کلریدریک) استفاده شد. روش مورد استفاده روش پلاریزاسیون پتانسیومتری می‌باشد. نتایج بدست آمده نشان داد که راندمان بازدارندگی با افزایش غلظت بازدارنده افزایش می یابد. منحنی‌های پلاریزاسیون نشان داد که این نوع بازدارنده از نوع مختلط می‌باشد. جذب سطحی مولکول‌های بازدارنده بر روی سطح استیل از ایزوترم لانگمویر تبعیت می کند. علاوه بر روش‌های الکتروشیمی،از روش آماری ارزیابی داده‌ها (مدل روش ماشین‌برداری حمایت‌کننده)برای اثبات اثر بازدارندگی و ساختار مولکولی بازدارنده استفاده شد.

واژه‌های کلیدی
بازدارنده خوردگی؛ ماشین‌برداری حمایت‌کننده؛ پلاریزاسیون پتانسیومتری.