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Thermodynamic Study of Corrosion Inhibition of Mild Steel in Corrosive Medium by *Piper nigrum* Extract

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Abstract

Background/Objectives: Introduction of green corrosion inhibitor can stop the use of expensive and harmful synthetic inhibitor. This study investigates the corrosion inhibition of mild steel in aggressive media by using *Piper nigrum* extract. **Methods/Statistical Analysis:** The potentiality of *Piper nigrum* to inhibit corrosion was studied based on concentration of inhibitors range of 0.2g/l-0.5g/l and temperature range of 40°C-60°C by using gravimetric and thermometric methods. **Results:** Calculation from weight loss method revealed that Inhibition Efficiencies (IE%) increase with increasing inhibitor concentration which show a decrease in Corrosion Rate (CR). However, the rise in temperature decreases the IE% which means an increasing in the CR. Furthermore, thermodynamic parameters of adsorption process such as Activation Energy (Ea), Enthalpy ($\Delta H°$) and Entropy ($\Delta S°$) were calculated and these values showed a good interaction. A rise of Ea values with increasing inhibitor concentration proved a physical adsorption mechanism is taking place. The $\Delta H°$ calculated proved that this is an endothermic process. **Conclusion/Application:** Results from this study showed that *Piper nigrum* was an attractive alternative to prevent corrosion as it shows the great inhibition efficiency.

Keywords: Corrosion Inhibitor, Gravimetric Analysis, Thermodynamic Study, Thermometric

1. Introduction

Mild steel is the most common metal used nearly in all industrial and domestic purposes. Mild steel is relatively inexpensive and possess metal properties that making it adequate for many uses especially in food, petroleum, chemical and electrochemical industries and power production. However, this metal undergoes deterioration where it is exposed to acidic medium like sulphuric acid and hydrochloric acid, which are normally used in industry for pickling and de-scaling of metals[1].

Corrosion is also known as the atmospheric oxidation of metals where oxygen combines with the metal and forms a new layer that can either be bad or good. Corrosion can cause deterioration in mechanical properties of metals. In case of iron, it is called rusting whereas for steel is tarnishing. Corrosion is an unfavourable as many direct and indirect costs arise due to the damages such as productivity losses interruptions, breakdowns, environmental pollutions and even some legal actions. Thought these effects are reversible and not an everlasting damages, it required a long time for thorough recovery.

Nowadays, there are many alternatives taken to prevent corrosion like choosing anti-corrosive materials in the real environment, design modifications to the system or components, electrochemical control, conditioning the metal and modify the environment either by removing the oxygen or adding inhibitors[2]. Among them, the use of inhibitor as corrosion inhibitor has become popular and gain attention due to its practical and applicable in many unit operation systems.

Corrosion inhibitor can be grouped into two categories, namely organic and inorganic green inhibitor at which organic green inhibitor shows higher inhibition efficiency compared to inorganic green inhibitor[3]. According to Acharya et al.[3], organic green inhibitors are

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the alkaloids and flavonoids and other natural products derived from natural sources that also contain trifling or negligible amount of toxicity. The inhibitor contains hetero atoms and suitable to be used as inhibitor due to they are inexpensive, biodegradable, non-toxic and give a least safety and health concerns[4]. The presence of hetero atoms (S, N, O) with free electron pairs, aromatic ring with delocalized π-electrons, high molecular weight of alkyl chains and the substituent group boost its inhibition efficiency[5]. Similarly, T.S. Franklin Rajesh et al.[6] found that hetero atoms in extracted inhibitor are very effective inhibitors against corrosion in acidic environments.

In this study, *Piper nigrum* seed extract was used for corrosion inhibition of mild steel in sulfuric acid solution. *Piper nigrum* is very suitable to be used as a corrosion inhibitor where it is a renewable resource and readily available in nature and even in the market. A rich source of alkaloid piperine like piperidine, piperettine and piperanine can be traced from the extract of black pepper[7]. Thus, the purpose of this study is to explore the potentiality of *Piper Nigrum* becoming a green corrosion inhibitor based on parameter of inhibitor concentration and temperature of acid medium.

2. Methodology

2.1 Preparation of Mild Steel Coupons

Mild steel test coupons were prepared by cutting mild steel plate into pieces with dimension of 3.0cm×2.0cm×0.2cm. Those specimens were then polished with different grades of emery paper from Grade Nos. 100, 150, 320 and 1200. After polishing the specimens, rinsed them with distilled water before rinsed again with acetone. Specimens were dried before kept sealed in zipper bag.

2.2 Plant Extraction

The fresh and dried black pepper seed, *Piper Nigrum* were purchased from an agriculture farm located in Shah Alam. The *Piper nigrum* were washed and rinsed with deionized water triplicate and dried. After it was completely dried, the *Piper nigrum* then was grinded by using grinder into a powder form. The extraction of the pepper was obtained by mixing 60 grams of ground pepper together with 4 grams of calcium carbonate in 500ml boiling flask. 400ml 2-propanol was added into the mixture and refluxed for 2 hours on water bath at 80°C before filtered and transferred into 500ml boiling flask. The 2-propanol was distilled off until left 20ml and transferred into 100ml beaker for crystallization of piperine.

2.3 Gravimetric Method

Fresh 100ml of 0.5M H_2SO_4 was prepared in a 200ml beaker and immersed mild steel coupon into it and added together 0.1g/l of *Piper nigrum* extract at temperature of 30°C by using 100µL micropipette. The coupon was weighed before the immersion and recorded. After 3 hours of immersion in incubator, the coupon was retrieved and rinsed with distilled water before weighed again. The procedures were repeated by using different extract concentration of 0.2, 0.3, 0.4 and 0.5g/l at temperature of 40°C, 50°C and 60°C. Every experiment was carried out duplicated.

The average weight loss of the mild steel coupon was taken as the difference between the Initial Weight (w_o) of the mild steel and Final Weight (w) after the immersion. This difference can be used to determine the Inhibition Efficiency (IE%) by using Equation 1.

$$IE\% = \frac{(w_o - w)}{w_o} \times 100\% \qquad (1)$$

Surface coverage (∅) is important in order to know interaction of inhibitor with aluminium surface[8] that can be determined through Equation 2.

$$\varnothing = \frac{(w_o - w)}{w_0} \qquad (2)$$

Corrosion Rate (CR) measures the effectiveness of inhibitor and directly related with weight loss in corrosive medium for estimated period in the unit of mmpy where W is the weight loss of mild steel (g), t is time exposure (h), A is the exposed area (cm^2) and ρ is the density of mild steel (g/cm^3).

$$CR = \frac{876\mathrm{W}}{\mathrm{At\rho}} \qquad (3)$$

Equation 4 was used to determine the activation energy (E_a) where R is universal gas constant, T is absolute temperature (K) and A is frequency factor.

$$\mathrm{E_a} \log \mathrm{CR} = \frac{-\mathrm{E_a}}{2.303\mathrm{RT}} + \log A \qquad (4)$$

Arrhenius equation (Equation 5) is used for thermodynamic study where N is Avogadro's number; h is the Planck's constant, ΔH° is enthalpy and ΔS° is entropy of activation. The plot of $\log \frac{\mathrm{CR}}{\mathrm{T}}$ versus $\frac{1}{\mathrm{T}}$ will give a straight line with slope of $\left[\frac{-\Delta H^\circ}{2.303R}\right]$ and intercept of $\left[\log R / Nh + \frac{\Delta S^\circ}{2.303R}\right]$ thus giving the values of ΔH° and ΔS°.

$$C_R = \frac{RT}{Nh}\exp\left(\frac{\Delta S^\circ}{R}\right)\exp\left(\frac{-\Delta H^\circ}{RT}\right) \quad (5)$$

2.4 Thermometric Method

A three-neck flask was used to carry out this method with thermometer inserted in 100ml of test solution of 0.5M H_2SO_4. A mild steel coupon prepared before was put into the flask until it was fully immersed. Add corrosion inhibitor of 0.1g/l, 0.2g/l, 0.3g/l, 0.4g/l and 0.g/l. The initial temperature of the solution is kept at room temperature. The change in temperature with time was measured by using digital thermometer. From the rise in temperature per minute, reaction number (RN) can be calculated from Equation 6:

$$RN\left(^\circ C\,min^{-1}\right) = \frac{T_\infty - T_i}{t} \quad (6)$$

where T_∞ and T_i are maximum and initial temperature (°C) respectively and t was time taken to reach the maximum temperature (min). The percentage reduction of reaction number (I%) can be calculated by using Equation 7:

$$I\% = \frac{RN_{aq} - RN_{wi}}{RN_{aq}} \times 100\% \quad (7)$$

where RN_{aq} and RN_{wi} were reaction number in the absence of inhibitor and reaction number in the presence of inhibitor respectively.

3. Results and Discussion

3.1 Gravimetric Analysis

The effectiveness of corrosion inhibition can be determine based on the IE%, CR and surface coverage (∅) that can be calculated by using formula from gravimetric method. The data on the study of corrosion inhibition efficiency of mild steel in 0.5M H_2SO_4 by using *Piper nigrum* extract are tabulated in Table 1(a), (b) and (c).

Table 1. **(a)** Data on the inhibition efficiency in 0.5M H_2SO_4 in the absence and presence of inhibitor for 3 hours immersion

C (g/L)	Inhibition Efficiency (IE%)			
	303 K	313 K	323 K	333 K
Blank	-	-	-	-
0.1	96.75	89.97	75.87	55.51
0.2	97.45	90.74	79.04	60.00
0.3	97.80	91.52	78.61	62.09
0.4	97.99	92.93	79.69	64.90
0.5	98.57	93.88	86.15	67.40

Table 1. **(b)** Data on the surface coverage (∅) in 0.5M H_2SO_4 in the absence and presence of inhibitor for 3 hours immersion

C (g/L)	Surface Coverage (∅)			
	303 K	313 K	323 K	333 K
Blank	-	-	-	-
0.1	0.9675	0.8997	0.7587	0.5551
0.2	0.9745	0.9074	0.7904	0.6000
0.3	0.9780	0.9152	0.7861	0.6209
0.4	0.9799	0.9293	0.7969	0.6490
0.5	0.9857	0.9388	0.8615	0.6740

Table 1. **(c)** Data on the corrosion rate in 0.5M H_2SO_4 in the absence and presence of inhibitor for 3 hours immersion.

C (g/L)	Corrosion Rate (CR) (mm/yr)			
	303 K	313 K	323 K	333 K
Blank	0.3391	0.9563	2.3062	4.3090
0.1	0.2923	0.8875	2.1113	3.9783
0.2	0.2288	0.8063	1.9491	3.8047
0.3	0.1925	0.7393	1.8651	3.5229
0.4	0.1770	0.5859	1.7694	3.0787
0.5	0.1252	0.5071	1.2036	2.8251

Generally, the IE% will increase as the corrosion inhibitor increase. The presence of inhibitor will provide a film barrier protecting mild steel surface from being exposed to acidic medium that can enhance the corrosion process. Increasing of IE% will reduce the rate of corrosion process due to higher surface coverage (∅) from inhibitors.

In this study, the maximum inhibition efficiency reach is 98.57% at solution of 303K with 0.5g/L of *Piper Nigrum* extract. This is due to the concentration of inhibitor is the highest and can protect the mild steel surface form the aggressive H_2SO_4 environment. At this condition, the surface coverage is the highest with 0.9857 compare to others. Large surfaces areas of mild steel are covered by inhibitor's film and this reducing the reaction sites available. As a result, this in return giving the least corrosion rate of 0.1252mm/yr due to the presence of inhibitor that provide film barrier to corrosive agents.

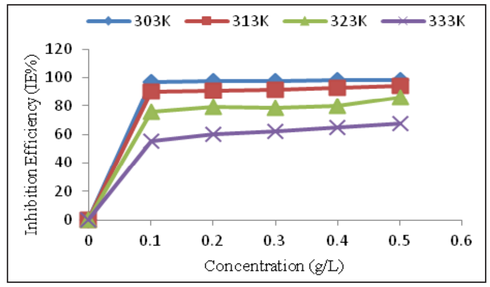
However, the least inhibition efficiency effect is recorded in 333K H_2SO_4 solution with 0.1g/L of *Piper nigrum* extract with 55.51%. The lower amount of inhibitor gives the less protection on mild steel surface thus exposed the metal to corrosive environment. More surface of mild steel available for corrosion process. The surface coverage at this point is 0.5551mm/yr, the least surface coverage compare to others. Corrosion rate for a blank solution is at the highest rate (4.3090mm/yr) with highest temperature (333K) due to there is no inhibitor available to provide a protection on mild steel surface from corrosive acid. The corrosion process occurred rapidly at higher temperature due to the hot-movement of inhibitor molecules that weakening the adsorption capacity on mild steel surface[1].

The relationship between concentration of inhibitor and temperature H_2SO_4 on IE% and CR can be further explained by using Figure 1(a) and (b). The same trend on IE%, CR and surface coverage (∅) was reported by earlier studies on corrosion inhibition of mild steel by using green inhibitors such as jujube leaves[1], tea[9], sunflower leaves[10] and banana peels[11]. According to Quraishi et al.[7], adsorption of inhibitors on mild steel surface will lead interaction between π-electrons of inhibitor molecules and vacant d-orbitals of mild steel surface atoms. Consequently barrier film will be formed between metal surface and corrosive medium.

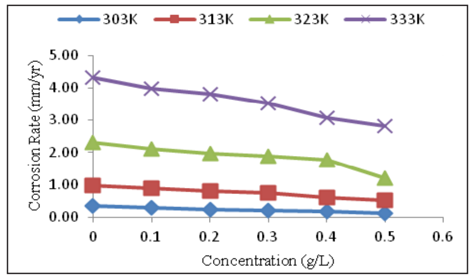
It was found out that IE% increased with ∅ as the concentration inhibitor increasing and decrease as temperature increase[8,12–14]. The same goes for CR that will decrease when IE% increasing and vice versa.

3.2 Thermometric Analysis

Thermometric analysis was carried out to determine the reaction number in this study. The values for RN and reduction of reaction number IE% of can be calculated by using Equations 6 and 7 respectively. Data on the calculated RN and percentage IE% of reduction number are tabulated in Table 2.



(a)



(b)

Figure 1. **(a)** Inhibition efficiency of mild steel in 0.5M H2SO4 in various concentrations (g/L) and temperature (K). **(b)** Corrosion rate of mild steel in 0.5M H2SO4 in various concentrations (g/L) and temperature (K).

Table 2. Reaction number and percentage inhibition efficiency of reduction number for mild steel dissolution in 0.5M H_2SO_4

Concentration (g/l)	Reaction Number (RN) (°Cmin^{-1})	Reduction in RN (Inhibition Efficiency, I%)
Blank	33.2692	-
0.1	28.3447	14.80
0.2	22.7273	31.69
0.3	21.5152	35.33
0.4	20.8768	37.25
0.5	18.9723	42.97

Data tabulated in Table 2 shows a higher I% at inhibitor concentration of 0.5g/l. This value corresponds for 18.9723°Cmin^{-1} that gives an inhibition efficiency of 42.97%. As concentration of inhibitor increases, more surface area of mild steel covered by inhibitor molecules thus preventing corrosion process. Therefore, the IE% also increases. This assertion also corroborated with gravimetric analysis.

3.3 Thermodynamic Studies

Thermodynamic properties such as Activation Energy (E_a), Enthalpy (ΔH°) and Entropy of Activation (ΔS°) are studied in order to identify the mechanism of adsorption process involved. The data for E_a, Δ°H and Δ°S involved in this study are tabulated in Table 3.

Based on Table 3, the values of activation are increases as concentration of inhibitor increases. The value of E_a in blank solution is 71.55kJ/mol and raises as concentration of inhibitors increases from 0.1g/l (73.20kJ/mol) to 0.5g/l (85.93 kJ/mol). This is due to the physical barrier created by adsorbed molecules on mild steel surface will increased the minimum energy required for corrosion reaction to occurs. More inhibitors will produce stronger film barriers.

The trend of increasing E_a values as with concentration inhibitors also been reported by earlier studies on various plant extract such as jujube leaves[1], black pepper[7,15], sunflower leaves[10], banana peels[16] and *Centella asiatica* leaves[17]. According to Quraishi et al.[7], the corrosion reaction will be push away on surface site and occurs at the uncovered parts of mild steel when inhibitors are added in acid solution thus giving a higher value of E_a.

Values of Activation Energy (E_a) are used in this study to determine the adsorption mechanism occurred. Zarrouk et al.[18] found that the constant or lower values of E_a in inhibited system compare to the blank solution shows a chemisorption. Despite the higher values of E_a attribute to physical adsorption mechanism. In this study, the increasing values of E_a clearly stated a physical adsorption of inhibitor molecules occurs. Physical adsorption happens due to the electrostatic force between negatively charged metal surface and positive charged of organic species.

Table 3. Values for Ea, ΔH° and ΔS° in the absence and presence of inhibitors with different concentration

C (g/l)	Activation energy (E_a) (kJ/mol)	Enthalpy (ΔH°) (kJ/mol)	Entropy of Activation (ΔS°) (kJ/mol.K)
0.0	71.55	68.91	-0.0261
0.1	73.20	70.56	-0.0217
0.2	78.43	75.79	-0.0062
0.3	81.26	78.62	0.0019
0.4	81.45	78.81	0.0015
0.5	85.93	83.29	0.0136

Enthalpy values (ΔH°) in corrosion inhibition process of *Piper nigrum* in 0.5M H_2SO_4 increases with increasing concentration inhibitors. The value of in blank solution is 68.91kJ/mol and increase to a maximum of 83.29kJ/mol at highest concentration of inhibitors (0.5g/L). This indicates the additional of inhibitors retard the corrosion process and more energy is needed for it to break the film barrier and react with mild steel surface. The positive values of ΔH° define an endothermic process of mild steel dissolution and it is difficult for it to dissolute[17].

The entropy value for uninhibited system is 0.0261kJ/mol.K and increasing as inhibitors added into the acid medium. The maximum value of ΔS° was reported at the inhibitor concentration of 0.5g/L with 0.0136kJ/mol.K. Observation on the trend of ΔS° shows the values are shifting toward positive values. This is indicate the activated complex in the rate determining steps show an association rather than dissociation steps, meaning that a decrease in disordering occurs on going from reactants to the activated complex[7,10,17,19]. The higher the concentration of inhibitor, the more associated the order of activated complex involved in the rate determining step.

Increasing inhibitor concentration will decrease the disorder of the system due to adsorption of inhibitor molecules on mild steel surface. In addition, small negative values of ΔS° for inhibited system showed a number of water molecules on mild steel surface being displaced by inhibitor molecules[4].

4. Conclusion

Corrosion inhibition process was carried out to retard corrosion process that creates many problems in human daily life. In this study, organic plant from of *Piper nigrum* (black pepper) extract was studied for their role as corrosion inhibitor due to the presence of certain compounds which attribute to their inhibition properties. It was observed that percentage IE% reach a maximum of 98.57% when inhibitor concentration is 0.5g/L due to higher surface coverage of extract on surface of mild steel (0.9857). However, the lowest inhibition efficiency with 55.51% was recorded at the lowest amount of inhibitor concentration, 0.1g/L, due to lower surface coverage of extract (0.5551). In addition, corrosion rate was reported at maximum rate in blank solution where there is no inhibitor to retard the deterioration process. The higher the concentration of inhibitor, the higher the percentage of IE% and the lower rate of corrosion. Therefore, it is clearly that concentration of inhibitor affecting the efficiency of inhibition process.

Temperature also affected much on the rate of corrosion and efficiency of *Piper nigrum* extract to inhibit corrosion process. Temperature of acid medium will offer better inhibition efficiency at low temperature compared to high due to the increasing in kinetic energy of molecules will increase the rate of reaction occurred. This explained highest percentage of inhibition efficiency occurred at 303K (98.57%) rather than 333K (67.40%) at concentration of 0.5g/L. From thermometric studies, a conclusion on the RN can be draw form the data collected. Reaction number was decreased as the concentration inhibitor increases thus giving an increment in the I% from 14.80% to 42.97%.

Adsorption mechanism of molecules on mild steel surface can be explained from thermodynamic analysis of Activation Energy (E_a), Enthalpy (ΔH°) and Entropy of Activation (ΔS°). Activation energy of the corrosion inhibition process increasing from 71.55kJ/mol to 85.93kJ/mol as temperature increases. The value of activation can be used to identify type of adsorption process involved whether chemisorptions or physical adsorption. It is proved that physical adsorption involved in this study as value of activation energy in inhibited system increases higher than in the blank solution. The same goes for enthalpy that rises from 68.91kJ/mol to 83.29kJ/mol. The positive values of enthalpy denoted an endothermic process. The values for entropy increases and shift toward positive values from -0.0261kJ/mol.K to 0.0136kJ/mol.K. Thus, from all the parameters being studied and analysis being carried out, results strongly indicate that *Piper nigrum* extract has been successfully acts as an effective corrosion inhibitor and can be used as an alternative method to prevent corrosion.

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