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# Multi-Factorial Evaluation of Corrosion Inhibition Efficiency of Bilberry Cactus Extract on Mild Steel in Hydrochloric Acid Solution

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**Abstract:** This paper presents multi-factorial evaluation of corrosion inhibition efficiency (due to weight loss) of bilberry cactus extract on mild steel in hydrochloric acid solution. Reaction between the mild steel and HCl during the corrosion process was believed to have resulted to hydrogen evolution. Hydrogen evolution rate was directly proportional to the mild steel corrosion rate. Furthermore, inhibition efficiencies due to weight loss and hydrogen evolution were evaluated to be directly proportional and almost equal. This implies that the mild steel corrosion was as a result of the hydrogen evolution. An empirical model; $\xi = V(\vartheta / \mathcal{S})^{0.0001}$ was derived to evaluate the corrosion inhibition efficiency (due to weight loss) of bilberry cactus extract on the mild steel. The validity of the model was rooted on the core model expression $(\mathcal{S} / \vartheta)^{0.0001} = V/\xi$ where both sides of the expression are correspondingly equal to unity. The standard error incurred in predicting the model-based inhibition efficiencies (due to weight loss) relative to the actual results is 0.8668. Deviational analysis of model-predicted results with respect to actual results was < 3%. This translated into over 97% operational confidence levels for the derived model. The correlation coefficients between inhibition efficiency (due to weight loss) and corrosion rate, hydrogen evolution rate & inhibition efficiency (due to hydrogen evolution) were all > 0.97.

**Key words:** Mild steel corrosion · Bilberry cactus extract · Hydrochloric acid

## INTRODUCTION

Structural failures resulting from corrosion of metals and alloys have been widely considered to bring about huge financial losses and uncertainties in the sustenance of chemical and metallurgical industries.

Research [1] has tipped mild steel as the most commonly used material in fabricating and manufacturing of oil field operating platforms due to its high strength, ease of fabrication, low cost and availability.

Mild steel are exposed to some industrial media such as elemental gases, inorganic salts and acid solutions, most of which influence corrosion rates [2], through processes such as cleaning of refinery equipment, pickling, oil well acidizing and acid descaling [3]. It is believed that the exposure could be so severe to the structural properties of the metals that sudden and catatrophic failures become inevitable.

Corrosion inhibition of metals in acidic solution involves attempting to reduce the concentration of corrosive species; $H^+$ present in the solution [4].

Corrosion inhibition of mild steel in 1 M $H_2SO_4$ by leaves and stem extract of Sida acuta was studied [5] using weight loss, hydrogen evolution and spectroscopic (AAS, FTIR and UV-V) techniques at a temperature range of 30-60°C. The results of the investigation reveal that Sida acuta leaves and stem extracts inhibited the acid induced corrosion of the mild steel. The inhibition efficiency increased with increase in the concentration of the extracts but decreased with rise in the reaction temperature. The inhibitive effect of the extracts was attributed to its adsorption on the mild steel surface as approximated by Freundlich adsorption isotherm.

Similar studies [6] carried out to evaluate the inhibition efficiency of pineapple leaves extract on mild steel in HCl using the same process parameters as in the research [5] indicated that pineapple leaves extract also

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inhibited acid induced corrosion of mild steel. Furthermore, inhibition efficiency increased with increase in the extract concentration. However, adsorption of the extract obeyed Langmuir adsorption isotherm at all concentrations and temperatures considered.

Another scientist [7] studied the corrosion inhibition characteristics of Hunteria umbellate seed husk extract on mild steel immersed in 1 M HCl and $H_2SO_4$ solutions. Scanning electron microscopy (SEM), atomic absorption spectroscopy (AAS), Fourier transform infra-red spectroscopy (FTIR) and mass loss were used as basis for studying the corrosion inhibition behavior of the extracts. Results of the study show that mild steel corrosion was significantly inhibited in both acidic environments. This was attributed to the O-H, C-N and unsaturated C=C functional groups present in the extracts. The research work also indicated that the inhibition efficiency increased with increase in the concentration of the extracts in the acidic solutions, but decreased with increase in the reaction temperature. The researcher concluded that the inhibition efficiency of the extracts was quite high but more effective in 1 HCl compared to 1 M $H_2SO_4$ solution.

Results of investigation [8] on the corrosion inhibitive effect of black pepper extract on the corrosion rate of mild steel in 1 $H_2SO_4$ solution indicated that the extract acted as good inhibitor even at high temperatures. The research was carried out using the conventional weight loss, Tafel polarization, impedance spectroscopy and Scanning electron microscopy (SEM) techniques. The polarization curves revealed the mixed mode of inhibition of black pepper extract.

Corrosion inhibition on mild steel in $H_2SO_4$ has also been studied using pomegranate alkaloid [9]. In this research, galvanostatic polarization and mass loss measurement were carried out at different temperatures. Results of the study showed that pomegranate alkaloid had reasonable corrosion inhibition efficiency at low temperatures. The researcher attributed this property to the metal additive complex formation.

The inhibitive effect of bilberry cactus extract on mild steel in 1.5 M HCl was studied [10] at a temperature and extract concentration range of 25-60°C and 0.1 -0.5 g/l respectively. This process was investigated using weight loss and hydrogen evolution measurement method. The results of the investigation reveal that the protective film formed on the surface of the mild steel was by adsorption of the extracts which obeyed Langmuir adsorption isotherm.

The aim of this research is to derive an empirical model for evaluating the corrosion inhibition efficiency (due to weight loss) of bilberry cactus extract on mild steel immersed in hydrochloric acid solution at different temperatures.

## MATERIALS AND METHODS

Materials used for the experiments are mild steel coupons (of composition; carbon 0.053%, silicon 0.14%, aluminium 0.2%, calcium 0.1%, manganese 0.48%, iron 97.48%, copper 0.057% and chromium 0.018%), 1.5M HCl solution (prepared by dilution of analytical grade 37% HCl with double distilled water), bilberry cactus plant, distilled water. The equipment used were pyrex beakers Micro drilling machine (Model H), analytical digital weighing machine (Mettle 4900) and pH meter. Other materials used and detailed experimental procedures are as shown in previous work [10].

## RESULTS AND DISCUSSION

Table 1: Variation of corrosion rate $\vartheta$ of mild steel in hydrochloric acid-bilberry extract solution with hydrogen evolution rate ʆ and inhibition efficiencies due to corrosion $\xi$ and hydrogen evolution Ѵ respectively [10]

| ($\xi$) | ($\vartheta$) | (ʆ) | (Ѵ) |
|---|---|---|---|
| 70.83 | 0.0036 | 0.0086 | 70.11 |
| 66.92 | 0.0043 | 0.0160 | 67.05 |
| 63.98 | 0.0051 | 0.0264 | 64.54 |
| 59.70 | 0.0059 | 0.0358 | 60.31 |
| 44.76 | 0.0086 | 0.0568 | 43.49 |

Computational analysis of the actual results shown in Table 1, gave rise to Table 2 which indicate that;

$$\left[\frac{ʆ}{\vartheta}\right] = \left[\frac{Ѵ}{\xi}\right]^{K} \quad (1)$$

Introducing the value of K into equation (1) reduces it to;

$$\xi = Ѵ \left[\frac{\vartheta}{ʆ}\right]^{K} \quad (2)$$

The derived model is equation (2).

where

K = 0.0001; equalizing constant (determined using C-NIKBRAN [11])

($\xi$) = Inhibition efficiency due to corrosion (%)

(Ѵ) = Inhibition efficiency due to $H_2$ evolution (%)

($\vartheta$) = Corrosion rate of mild steel ($gcm^{-2}\ h^{-1}$)

(ʆ) = Hydrogen evolution rate ($ml\ cm^{-2}\ min^{-1}$)

Table 2: Variation of $V/\xi$ with $(ẞ/\vartheta)^K$

| $V/\xi$ | $(ẞ/\vartheta)^K$ |
|---|---|
| 0.9898 | 1.00090 |
| 1.0019 | 1.00013 |
| 1.0088 | 1.00016 |
| 1.0102 | 1.00018 |
| 0.9716 | 1.00019 |

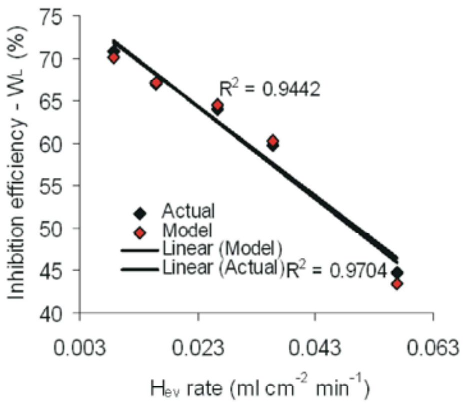


Fig. 1: Coefficient of determination between inhibition efficiency due to weight loss and hydrogen evolution rate as obtained from actual and model-predicted results

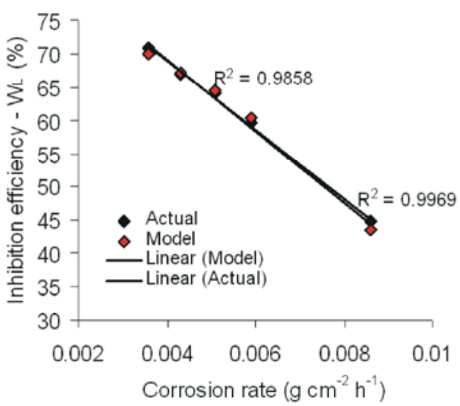


Fig. 2: Coefficient of determination between inhibition efficiency due to weight loss and corrosion rate as obtained from actual and model-predicted results

**Boundary and Initial Conditions:** Consider short cylindrically shaped mild steel coupon submerged in hydrochloric acid-bilberry cactus extract solution, interacting with some corrosion-induced agents. The solution is assumed to be affected by undesirable dissolved gases. The considered range of the solution temperatures, corrosion rate, hydrogen evolution rate, inhibition efficiencies due to weight-loss and hydrogen evolution are 25-60 (°C), 0.0036-0.0086 (g $cm^{-2}$ $h^{-1}$), 0.0086-0.0568 (ml $cm^{-2}$ $min^{-1}$), 44.76-70.83 (%) and 43.49-70.11 (%) respectively.

**Model Validity:** The validity of the model is strongly rooted on the core model equation (1) where both sides of the equation are correspondingly equal to unity. Table 2 also agrees with equation (1) following the values of $(ẞ/\vartheta)^K = V/\xi$ evaluated from the actual results in Table 1. Furthermore, the derived model was validated by comparing the inhibition efficiency (due to weight loss) predicted by the model and that obtained from the experiment. This was done using various analytical techniques which includes statistical, graphical and deviational analyses.

**Statistical Analysis**

**Correlation:** The correlation coefficient between inhibition efficiency (due to weight loss) and corrosion rate, hydrogen evolution rate & inhibition efficiency (due to hydrogen evolution) were evaluated (using Microsoft Excel Version 2003) from results of the actual and derived model. These results are 0.9984 and 0.9929, 0.9851 and 0.9717 & 0.9975 and 1.0000 respectively. The evaluations were based on the coefficients of determination $R^2$ shown in Figs. 1-3 and calculated using equation (3).

$$R = \sqrt{R^2} \tag{3}$$

**Standard Error (STEYX):** The standard error incurred in predicting the model-based inhibition efficiencies (due to weight loss) relative to values of the actual results following substitution of the essential process parameters; corrosion rate, hydrogen evolution rate & inhibition efficiency (due to hydrogen evolution) into the model equation is 0.8668. The standard error was evaluated using Microsoft Excel version 2003.

**Graphical Analysis:** Comparative analysis of Figs. 4-6 show curves and shapes perimeters of model-predicted and actual results with high degree of alignment. This indicates proximate agreement between both results.

**Deviational Analysis:** Analysis of the inhibition efficiency (due to weight loss) precisely obtained from the actual and model-predicted results shows single digit deviation on the part of model-predicted results. This was attributed to the fact that the effects of the surface properties of the mild steel which played vital roles during corrosion in hydrochloric acid- bilberry cactus extract solution were not considered during the model formulation. This necessitated the introduction of correction factor, to bring the model-predicted inhibition efficiency (due to weight loss) to those of the corresponding experimental values.

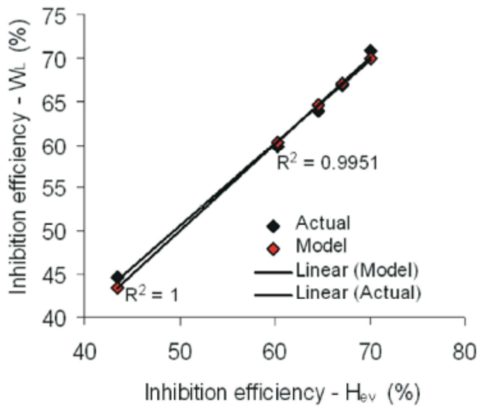


Fig. 3: Coefficient of determination between inhibition efficiency due to weight loss and inhibition efficiency due hydrogen evolution as obtained from actual and model-predicted results

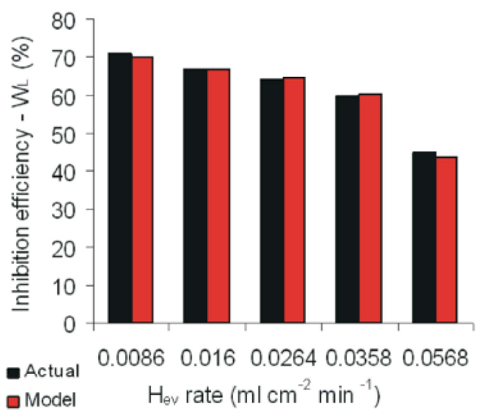


Fig. 4: Variation of inhibition efficiency due to weight loss with hydrogen evolution rate as obtained from actual and model-predicted results

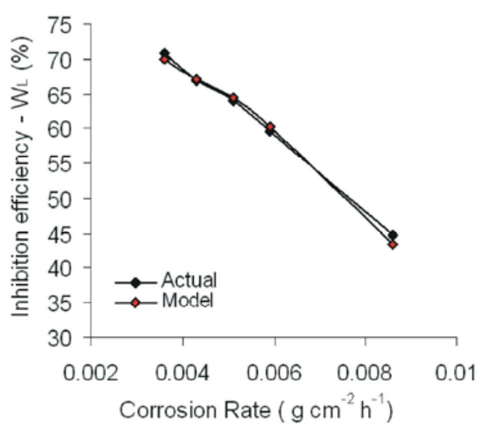


Fig. 5: Variation of inhibition efficiency due to weight loss with corrosion rate as obtained from actual and model-predicted results

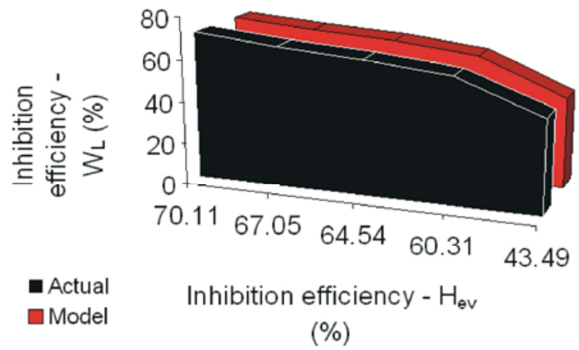


Fig. 6: Variation of inhibition efficiency due to weight loss with inhibition efficiency due hydrogen evolution as obtained from actual and model-predicted results

The deviation Dv, of model-predicted inhibition efficiency (due to weight loss) from the corresponding actual result was given by;

$$Dv = \left(\frac{\zeta_P - \zeta_E}{\zeta_E}\right) \times 100 \qquad (4)$$

where,

$\zeta_E$ and $\zeta_P$ are inhibition efficiencies (due to weight loss) evaluated from experiment and derived model respectively.

Figure 7 shows that maximum deviation of model-predicted inhibition efficiencies (due to weight loss) from the actual results was less than 3%. This translates into over 97% model operational confidence. The figure shows that the least and highest deviations of model-predicted results (from actual results) are 0.18 and – 2.86 %.

These deviations correspond to model-predicted inhibition efficiencies (due to weight loss): 67.0413 and 43.4817 %; corrosion rates: 0.0043 and 0.0086 g $cm^{-2}$ $h^{-1}$, hydrogen evolution rates: 0.0160 and 0.0568 (ml $cm^{-2}$ $min^{-1}$) and inhibition efficiencies (due to hydrogen evolution): 67.05 and 43.49 % respectively.

Correction factor, Cr to the model-predicted results was given by;

$$Cr = -\left(\frac{\zeta_P - \zeta_E}{\zeta_E}\right) \times 100 \qquad (5)$$

Comparative analysis of Fig. 7 and Table 3 shows that the evaluated correction factors are negative of the deviation as shown in equations (4) and (5).

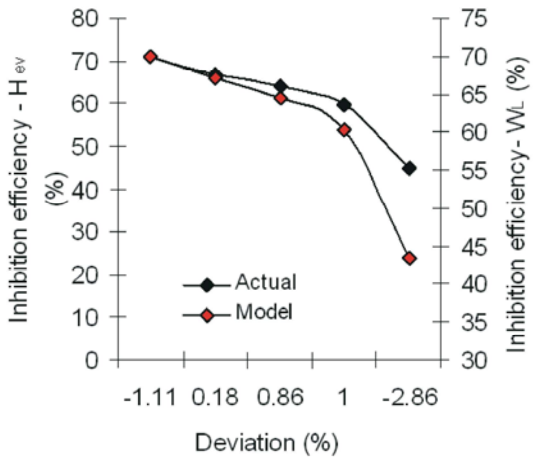


Fig. 7: Deviation of model–predicted results from actual values

Table 3: Variation of correction factor to model- predicted inhibition efficiency (due to weight loss) with the solution temperature

| Temp (°C) | Cr (%) |
|---|---|
| 25 | + 1.11 |
| 30 | - 0.18 |
| 40 | - 0.86 |
| 50 | - 1.00 |
| 60 | + 2.86 |

The correction factor took care of the negligence of operational contributions of the effects of surface properties of the mild steel which actually affected the corrosion process. Introduction of the corresponding values of Cr from equation (5) into the model gives exactly the corresponding actual inhibition efficiency (due to weight loss). Table 3 indicates that the maximum correction factor to the model-predicted inhibition efficiency (due to weight loss) was less than 3%. The table shows that the least and highest correction factors to the model-predicted results (from actual results) are - 0.18 and 2.86 %. These deviations correspond to model-predicted inhibition efficiencies (due to weight loss): 67.0413 and 43.4817 %; corrosion rates: 0.0043 and 0.0086 g $cm^{-2}$ $h^{-1}$, hydrogen evolution rates: 0.0160 and 0.0568 (ml $cm^{-2}$ $min^{-1}$) and inhibition efficiencies (due to hydrogen evolution): 67.05 and 43.49 % respectively.

The deviation of model predicted results from that of the actual is just the magnitude of the value. The associated sign preceding the value signifies deviation deficit (negative sign) or surplus (positive sign).

## CONCLUSION

Following multi-factorial evaluation of corrosion inhibition efficiency (due to weight loss) of bilberry cactus extract on mild steel in hydrochloric acid solution, reactions between the mild steel and HCl during the corrosion process resulted to hydrogen evolution. Hydrogen evolution rate was directly proportional to the mild steel corrosion rate. Furthermore, inhibition efficiencies due to weight loss and hydrogen evolution were directly proportional and almost equal. This implies that the mild steel corrosion was as a result of the hydrogen evolution. An empirical model; $\xi = V(\vartheta / \S)^K$ was derived to evaluate the corrosion inhibition efficiency (due to weight loss) of bilberry cactus extract on the mild steel. The validity of the model was rooted on the core model expression $(\S / \vartheta)^K = V/\xi$ where both sides of the expression are correspondingly equal to unity. The standard error incurred in predicting the model-based inhibition efficiencies (due to weight loss) relative to the actual results is 0.8668. Deviational analysis of model-predicted results with respect to actual results was < 3%. This translated into over 97% operational confidence levels for the derived model. The correlation coefficients between inhibition efficiency (due to weight loss) and corrosion rate, hydrogen evolution rate & inhibition efficiency (due to hydrogen evolution) were all > 0.97.

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