Global Advanced Research Journal of Engineering, Technology and Innovation (ISSN: 2315-5124) Vol. 5(3) pp. 034-039, July, 2016
Available online http://garj.org/garjeti/index.htm

Full Length Research Paper

Factorial Design Approach to Investigation of the Effect of *Blighia Sapida* Extract on Weight Loss of Pickled Mild Steel

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Accepted 23 May 2016

Mild steel undergoes reducible loss in weight during acid pickling operation in Electroplating Shop situated at SEDI-Enugu, Nigeria. The loss in weight was monitored and inhibited by acid extract of Blighia Sapida Pod .The effect of the extract was optimized by application of 2^3 factorial designs and optimum conditions were recorded at acid concentration of 4.41%w/v, pickling time of 1.55hrs and inhibition concentration of 5.36 g/l.The study more so revealed that increase in concentrations of the Blighia Sapida extract reduced greatly the weight losses in 0.95(%w/v) and 4.8(%w/v) concentration of hydrogen tetraoxosulphate (VI) solution respectively.

Keywords: *Bligha Sapida*, Steel, Factorial Design, Inhibitor Concentration, weight loss.

INTRODUCTION

Fabrication works and other heat treatment operations that occur at high temperatures usually leave a discolouring oxide layer, scale, and welding splash on the surface of mild steel. These surface deposits, otherwise known as impurities affect the electro deposition of nickel on the steel during plating operation. To remove the impurities, steel is dipped in a bath of pickling liquor where it experience fractional weight loss due to electrochemical reaction between its matrix and the interacting pickling liquor.

The benefit of this pickling operation is much rewarding save only the accompanying problem of weight loss and hydrogen embrittlement. These problems mentioned but few can be controlled by application of pickling inhibitors. Inhibitors generally, operate in three different descriptions: The film forming inhibitors which form a thin layer over the base metal to stop the reaction; the cathodic inhibitors which slow the reaction kinetics of the cathodic reaction and lastly the anodic inhibitors which slow anodic reaction.

Table 1. Experimental factors and levels for investigation on effect of *blighia sapida* extract.

FACTOR	LEVELS	
	Low Level(-)	High Level(+)
Inhibitor Conc. (g/l)	1.20	6.00
Acid Concentration. (%w/v)	0.95	4.80
Time (hrs)	1.00	7.00

Table 2. Factorial design showing treatment combination

Randomized Order	Run Order	Inhibitor Conc.(g/l)	Time (Hrs)	Acid Conc. (%w/v)	Response (gm)
5	1	1.2	1	4.8	0.560
8	2	6	7	4.8	0.272
1	3	1.2	1	0.95	1.360
4	4	6	7	0.95	0.487
6	5	1.2	7	4.8	2.288
8	6	6	1	4.8	0.058
3	7	6	1	0.95	0.190
2	8	1.2	7	0.95	2.883

In a pickling process, addition of these pickling inhibitors in a mild concentration can control weight loss to a certain degree of efficiency. Efficiency depends on pickling time, the type of metal, type of acid used, acid concentration, inhibitor concentration and pickling temperature. For higher efficiency and improved reduction in weight loss during pickling processes, the choice of pickling inhibitors is not overemphasized. Pickling inhibitors are selected either from synthetic compounds that have hetero atoms in their aromatic ring system or synthesized as extract from plant. Plant extracts constitute several organic compounds which have inhibiting abilities. The yield of these compounds vary widely depending on the part of the plant [Okafor, Ikpi, Ekanem, and Ebenso,(2013)]The present study is aimed at investigating the effect of *blighia sapida* extract on weight loss of pickled steel.

THEORITICAL BACKGROUND

Metals tend to return to the lowest energy state possible by means of corrosion [Mika,(2014)]. This tendency for metals to assume its lowest energy state most times occurs in a non-inhibiting pickling process. To understand corrosion mechanism in a non inhibiting pickling bath, the basic concepts of corrosion need to be reviewed with mild steel as a case study. Mild steel corrosion occurs by electrochemical reaction which requires an anode, a cathode, an electrolyte, and a circuitry path connecting the anode and the cathode. Mild steel corrodes when it interacts with water system, acidic medium, basic medium, salt, and corrosive vapours. When steel is exposed to any of the corrosive environment, it gives up electron, and become a positively charged ion. Oxidation of steel matrix occurs at the anode where the current enters the electrolyte. The anodic reaction would be: [Ejindu-Ejesi et al, 2016]

Anodic Reaction:

$$Fe \longrightarrow Fe^{2+} + 2e^{-} \quad \ldots\ldots (1)$$

Also, at the cathode end, reduction of oxygen occurs as described below:

Cathodic Reaction:

$$O_2 + 2H_2O + 4e^{-} \longrightarrow 4(OH^{-}) \quad \ldots\ldots (2)$$

Considering that there is no net gain of electron or loss of electrons, two atoms of iron dissociate to give four electrons needed at the cathode. Thus, the anodic and cathodic reactions would be:

$$2Fe \longrightarrow Fe^{2+} + 4e \text{ (anodic)} \quad \ldots\ldots (3)$$

$$O_2 + 2H_2O + 4e^{-} \longrightarrow 4(OH^{-}) \text{ (cathodic)} \quad \ldots\ldots (4)$$

These can be summed to give the overall oxidation-reduction reaction

Table 3. Analysis of variance [Partial sum of squares - Type III]

Source	Sum of Squares	Degree of freedom	Mean Square	F values	P values
Model	7.71	4	1.93	38.69	0.0065
A-Time	1.77	1	1.77	35.50	0.0095
B-Inh. Conc	4.63	1	4.63	92.84	0.0024
C-acid Conc	0.38	1	0.38	7.61	0.0702
AB	0.94	1	0.94	18.83	0.0226
Residual	0.15	3	0.050		
Cor Total	**7.86**	**7**			

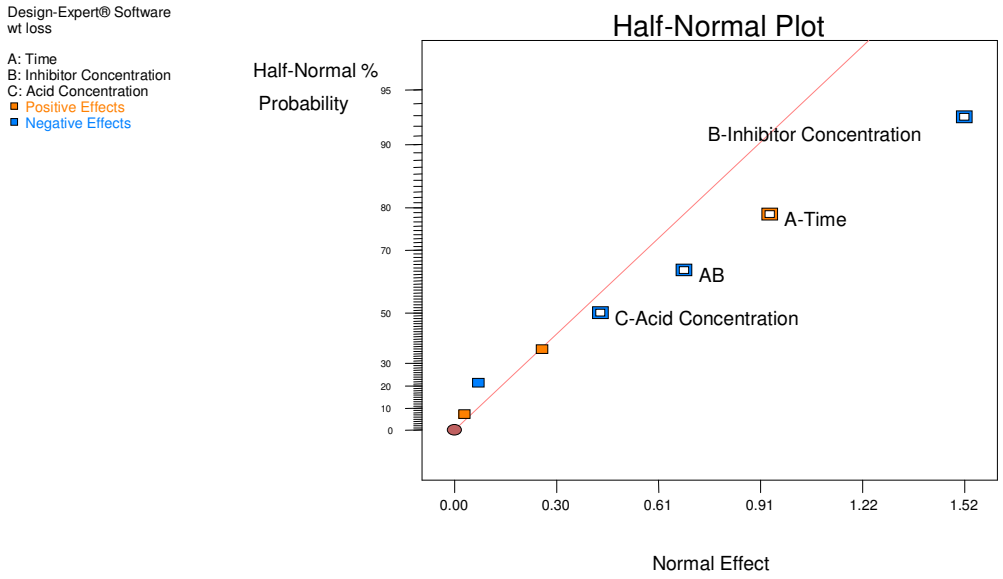

Figure 1. Half-normal plot of the effect of the factors.

$$2Fe + O_2 + 2H_2O \longrightarrow 2Fe^{2+} + 4(OH^-) \ldots\ldots (5)$$

MATERIALS AND METHODS

Weight Loss Experimental

The mild steel sheet was mechanically pressed cut into test coupons of measurement 3 × 4 × 0.01 cm. Each coupon was weighed on an analytical balance to 0.001g, degreased in absolute ethanol, dried in acetone, and preserved in desiccators for use. Also, fresh samples of B*lighia Sapida* fruit gotten from SEDI- Enugu Orchard were washed under running water, shade dried and cut to separate out into the seed, the pod, and the arils. The pod which was the plant part needed was sun dried, ground into powder and the powder obtained from the pod was weighed out into 1.2g, 2.4g, 3.6g, 4.8g, 6g. Each measured weight in gram was soaked in 100ml of 0.95 %(w/v) of H_2SO_4 and 4.8 %(w/v) of H_2SO_4 respectively for 24 hours. The soaked solutions were filtered in batches and the filtrates so obtained were used as the pickling solutions. The coupons preserved in the desiccators were pickled in several batches of 0.95 %(

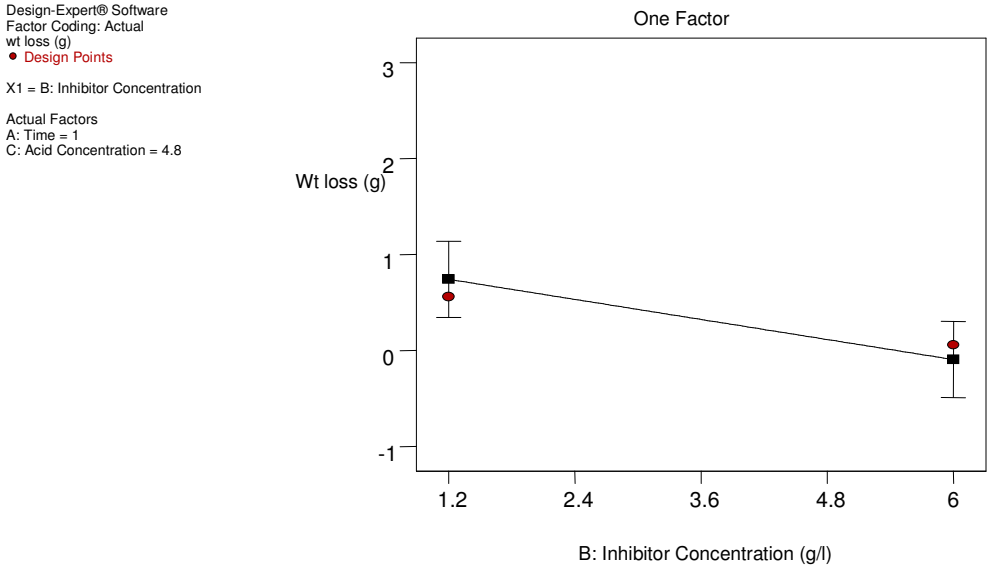

Figure2. One factor plot for the main effect of inhibitor concentration

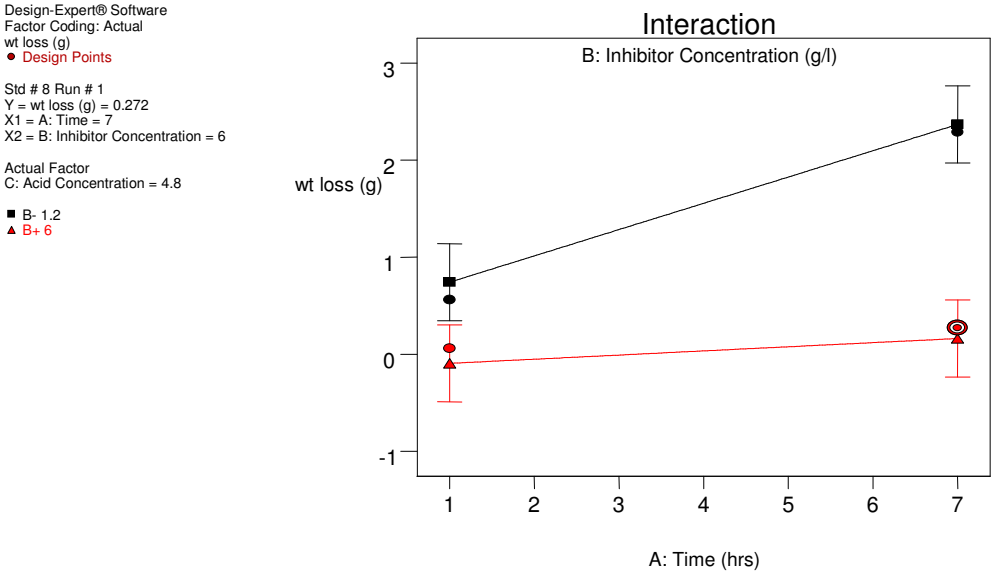

Figure 3 Interaction plot between inhibitor concentration and time

w/v) and 4.8%(w/v) solutions of H_2SO_4 (pickling solutions) holding varying concentrations of acid extract of *blighia sapida* for 7 hours

Factorial design

The experiment was conducted to investigate the effect of *blighia sapida extract* on reducing *the* weight loss of mild steel in the acid pickling process. The process variables considered for the investigation are time, acid concentration and inhibitor concentration. Two levels of each of the three factors were used for the statistical analysis. The treatment combinations for the two levels and three factors are tabulated in table 1 and 2

RESULTS / FINDINGS

Table 3 shows the result of ANOVA report of the selected model. The model F-value of 38.69 implies the model is significant. The values of P values less than 0.0500

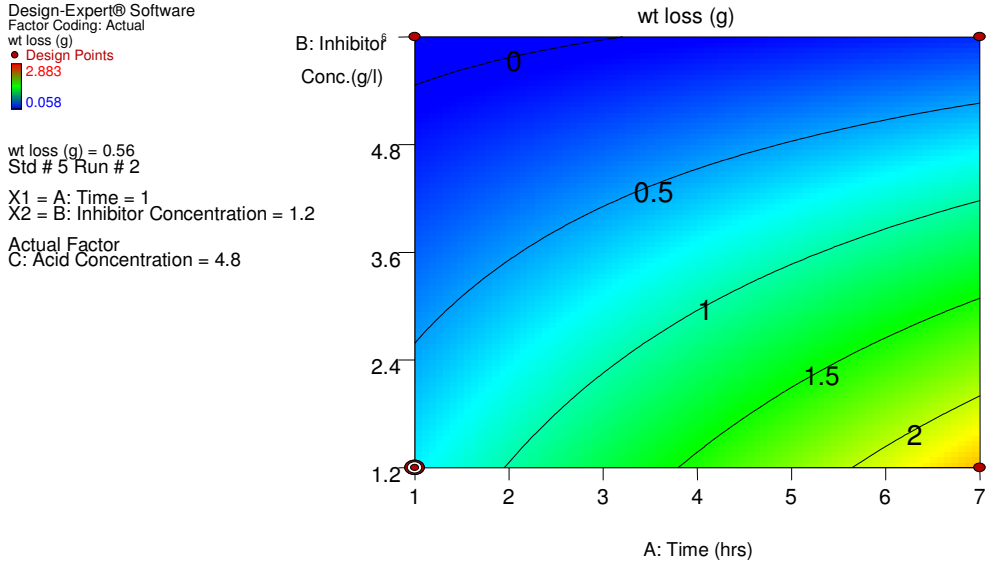

Figure 4 Contour plots between inhibitor concentration and weight loss.

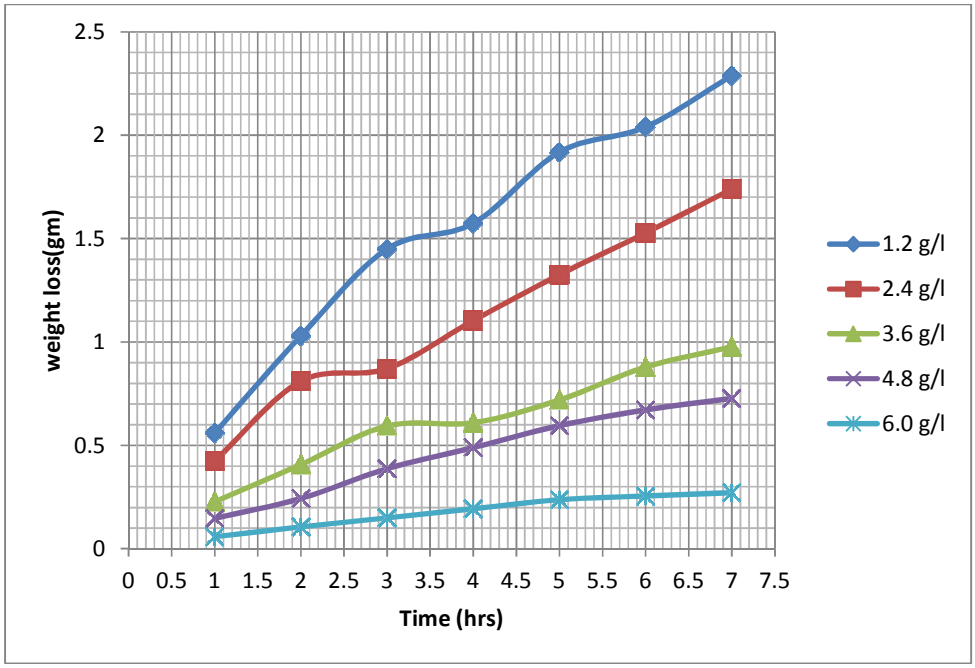

Figure 5. Weight loss plot at 4.8 (%w/v) H_2SO_4

indicate model terms are significant. In this model A, B and AB are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. The Predicated R-Squared is 0.8648 which is in reasonable agreement with the Adjusted R-Squared of 0.9556 since the difference between the predicated R^2 and the adjusted R^2 is less than 0.2. Adequate precision measures the signal to noise ratio. A ratio greater than 4 is desirable. The calculated ratio is 16.414 which indicate an adequate signal. The factorial model describing the process in terms of the denoted factors (A, B, C, and AB) is expressed below:

Wt. loss = 1.166 + 0.328A - 0.127B - 0.113C - 0.048A*B……..(6)

Where A, B and C are Time and Inhibitor concentration, acid concentration respectively

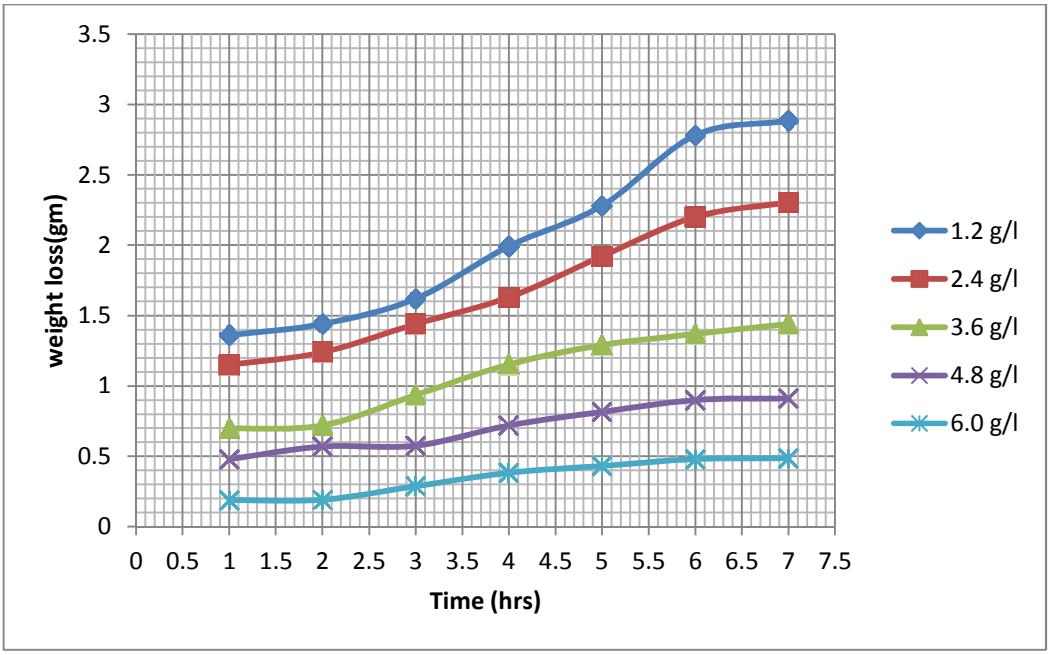

Figure 6. Weight loss plot at 0.95(%w/v) H_2SO_4

DISCUSSION

Figure 1 shows Half-Normal plot of the factors. From the Figure, the effect of Inhibitor Concentration B (1.52) is much more significant than the effect of time A (0.94).It means that the influence of inhibitor concentration on the final response was higher. The factors A and B were understudied to see how they interacted as (AB) to affect the weight loss of mild steel in the pickling solution.

Figure 2 shows the one-factor plot indicating the main effect of inhibitor concentration. From the plot it can be deduced that increase in inhibitor concentration resulted in the decrease in weight loss. The decrease in weight loss is shown by the steeply downward line plot.

Figure 3 shows the non parallel line plots that depicts how inhibitor concentrations and time interact to affect the weight loss. When the inhibitor concentration is low as represented by the black line, the weight loss increased steeply. However, when the inhibitor concentration is high as shown with the flat red line, the steel weight loss is greatly reduced. The effect of time on the weight loss also depends on the level of inhibitor concentration. At high inhibitor concentration (the red line plot), the weight loss is not significantly affected by time. At low inhibitor concentration (the black line), the effect of time on weight loss is significant.

Figure 4 below shows the tri-contour-plot between the inhibitor concentrations, time and weight loss. From the plot, it is observed that the inhibitor concentration increased with a continuous spread in the scalar value of the contours from the maximum value (2) to the minimum value (0).This indicate a significant decrease in weight loss.

Figure 5 and Figure 6 show graphical plots of weight loss at 4.8 (%w/v) H_2SO_4 and weight loss at 0.95(%w/v) H_2SO_4 respectively. Both plots highlighted the varying concentrations of the extract with their responsive effects on the weight loss over time.

CONCLUSIONS

The effect of *Bligha Sapida* on weight loss of pickled mild steel was studied under various design factors: time, inhibitor concentration and acid concentration. The result showed that the extract of *Blighia Sapida* reduced the weight loss of the steel. More so varying the concentration of *Blighia Sapida* extract from 1.2g/lit to 6.g/lit in the pickling liquor showed a significant reduction of the weight loss. The optimum values of the design factors - the optimum inhibition concentration, acid concentration and time - were 5.36 g/l, 4.41%w/v and 1.55 hrs, respectively.

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