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Inhibition of Corrosion of mild steel in 2M HCl by Determination of optimal experimental parameters using Factorial Design - A preliminary study

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Abstract : A preliminary study to investigate the effect of Pongamia minnata seed oil on the errosion inhibition of MS by weight loss method. This was increased by the applying 2^3 factorial design. The communicating possessions of concentration, reaction time, and temperature on inhibition were investigated; the input factors and output response were also optimized. Maximum inhibition efficiency of 94.27 % was achieved at low temperature of 27 ^{0}C, reaction time of 4 days and inhibition concentration of 10 %v/v. A combination of statistical analysis, the Pareto chart and normal possibility chart and the main effects and the interaction effects has been employed to obtain an in depth understanding of the corrosion variables. Analysis of variance on the corrosion constraint shows the fitness of this model. It can be determined that factorial design is adequately relevant in the optimization of process variables and the inhibitor Karanj (Pongmia pinnata) seed oil, adequately reserved the errosion of MS(mild steel) at the given surroundings of the investigation.
KeyWords : Factorial, Karanj.

1.0 Introduction

Pongamia pinnata is a class of family Leguminasae, inborn in tropical and temperate Asia. Commonly called as karanja. Karanja is dearth resistant, semi-deciduous, nitrogen fixing leguminous tree. It grows about 15-20 meters in height with a large canopy which spreads equally wide. The leaves are soft, shiny burgundy in early summer and mature to a glossy, deep green as the season progresses. It has various excellent therapeutic possessions. Its wood is used as fuel and various useful purposes. Its oil is a thick yellow – orange to brown extracted from seed. The oil is being used as fuel for cooking and lamps, as oil, insect killer and in detergent making industries. The crude oil is being analyzed as biodiesel[1].

In this work, Pongamia pinnata oil has been used to study the errosion inhibition of MS in acid medium. A full factorial design is used for the experiments and a polynomial equation is developed to relate the inhibition efficiency to the variables such as inhibitor concentration, time and temperature. The validity of the model is evaluated by the analysis of variance

2.0 Experimental

Pongamia pinnata seeds were cleaned, deshelled and dried at high temperature. The oil is extracted by soxhlet extraction method. Separated oil was dried over anhydrous sodium sulphate[7]. The physicochemical properties of karanja oil was performed according to the standard methods of AOAC, 1998 are shown in the table-1.

Table-1: Physicochemical analysis of Pongamia pinnata seed oil

Sl. No.	Parameters	Values
1	Specific Gravity	0.921
2	Water Content %	0.0498
3	Carbon Residue	0.79
4	Ash content %	0.05
5	Iodine Value	86.46
6	Saponification value	84.9
7	Flash Point (^oC)	212
8	Cetane Number	38

All corrosion inhibition data were obtained through weight loss experiments based on mild steel of surface area 5x1cm^2. The blank solution was 2M HCl. From the stock solution of the extract, different concentrations of the inhibitor test solutions ranging from 2% to 10 % v/v were prepared[1]. The samples in triplicate were immersed in 2M hydrochloric acid solution containing various absorptions of the inhibitor for 24 hours and 96 hours at 303 and 313 K. The samples were removed washed with water and dried. The physique of the samples earlier and later dipping was determined using an electronic digital balance[4].The errosion rate for various concentrations of inhibitor was obtained from the formula[2,3].

3.0 Factorial Design

A full factorial design with three variables temperature, concentration and time was used for the experiments. The experiments were conducted as per standards to investigate the given parameters affecting significantly the errosion rate and also the autonomously manageable chief process parameters. 2 stages of each of the 3 aspects were used for the statistical investigation. The treatment combinations for the two levels and 3 factors are tabulated in Table-2. The small & great heights for the aspects stayed nominated bestowing to some primary tests. The direction in which the investigations were made was randomized to avoid orderly errors. A full factorial design with three variables [Amount of inhibitor % v/v, Reaction time (minutes) and temperature (k)] is shown in the table-3. The optimum values of the variables were calculated with MINITAB 15

Table -2 Factorial design of the corrosion process showing treatment combination

Variables	Actual value		Coded value	
	Low level	High level	Low level	High level
Inhibitor (%v/v) (A)	2	10	-1	+1
Reaction time (days) (B)	1	4	-1	+1
Temperature ^0C (C)	27	30	-1	+1

Table-3- 2^3 full factorial design with statistical parameter design and the results

Inhibitor concentration (%v/v)	Reaction time (days)	Temp.(^{0}C)	IE (%) Trial-1	IE (%) Trial-2	IE (%) Trial-3	Average Inhibition Efficiency (%)
2	1	27	55	56.21	54.56	55.26
10	4	27	94.81	95.22	94.12	94.72
10	4	30	74.23	74.76	74.54	74.51
10	1	30	66.65	66.72	66.87	66.75
10	1	27	86.75	87.21	86.54	86.83
2	4	30	43.12	43.23	42.75	43.03
2	1	30	34.71	35.12	35.67	35.17
2	4	27	63.85	63.01	62.76	63.21

A mathematical expression to describe the design matrix combination mentioned in table-1 as low and high level of each factor and its corresponding corrosion rates mentioned in table-3 in code :corrosion Rate = f (A,B,C) Where A is inhibitor concentration, B is the time and C is the temperature[4]. The above model contains the properties of main variables1st order and 2nd order connections of all variables. Therefore the general model equation is given as[7][7] :

Corrosion Rate = β_0 + β_1*A + β_2*B + β_3*C + β_4*AB + β_5*BC + β_6*CA + β_7 *ABC

Where β0 is the average response of corrosion Rate and β_1, β_2 β_3, β_4, β_5, β_6 and β_7 are coefficients associated with each variables A, B, C and interactions. The deterioration coefficients and the accompanying affects are shown in tables-4. The significant factors are known by analysis of variance method. Based on the investigational results, a various linear regression model is developed and the effect of 95 % sureness levels for the extract is accessible in the table 4.

Table-4: Statistical parameters for 2^3 design

Factor	Degree of freedom	Coefficient	Effect
Average		65.00	
Inhibitor Concentration	1	16.00	32.00
Time	1	3.75	7.50
Temperature	1	-10.25	-20.50
Inhibitor concentration *Time	1	-0.25	-0.50
Inhibitor concentration * Temperature	1	0.25	0.50
Time * Temperature	1	2.50	5.00
Inhibitor concentration * Temperature * time	1	-0.00	-0.00

A reversion produced launches connection among the important positions attained from analysis of variance, namely temp, inhibitor and reaction time on the corrosion rate were statistically significant. Substituting the coded values of the variables at a given experimental conditions in the above equation the corrosion rate vales for the corrosion control behavior of the mild steel can be calculated. The developed model equation is expressed as

Corrosion Rate = 65.00 +32.00 A +7.50 B -20.50 C – 0.50AB +0.50 BC +5.00 AC -0.00*ABC

The above equation has been used to predict the errosion rate of the MS. The results of the linear regression model table -4 for pongamia oil displayed the inhibitor is the maximum significant variable with the key consequence of +32.00 miles per year followed by temperature (C) with -20.50 mpy and time B with +7.50 mpy. The deterioration discovered that temperature negatively impacted the inhibition efficiency of Pongamia seed oil on MS. The influence of inhibitor concentration was most pronounced as seen from the value of coefficient of that variable in comparison with the other . It can be resolved that once the outcome of a factor is positive an growth in the cost of the inhibition competence is detected when the factor changes from small to great level. In difference, if the effect is undesirable, a decrease in inhibition competence happens for great level of same issue.

3.1 Analysis of Variance

The results of analysis of variance are obtainable in the table-5. The analysis was estimated for a assurance level of 95 % for significance level of $F_{0.05}$=5.59,all possessions giving F greater than 5.59 have statistical meaning. The F –value for all models was more than 0.05, except for interactions. From the table-6 it is observed that the temperature (C), inhibitor (A) and time (B) are significant model positions inducing errosion rate of MS, since they have obtained F- value more than 0.05. Although the interaction effect was measured statistically irrelevant since their F- values are less than 0.05.

Table- 5 Analysis of variance-full fitting model for Castor seed oil

Factor	Sum of squares	Degree of freedom	Mean square	F value	P -value
Inhibitor concentration	2048.0	1	2048.0	9.19	9.19
Time	112.5	1	112.5	0.51	0.517
Temperature	840.5	1	840.5	20.63	20.63
Interactions					
Inhibitor concentration *Time	0.5	1	0.5	0.00	0.964
Inhibitor concentration *Temperature	0.5	1	0.5	0.03	0.877
Time * temperature	50.0	1	50.0	0.00	0.770
Residual	0.00	2	311.200		
Total	3052.00	7			

The various parameters selected for the validation test are revealed in the table -6.The effects of the validation tests stood found and comparisons were ended among the actual corrosion rate ideals and projected values[6-7]. The model predicts much more increase in inhibition efficiency increase with high and low levels of A, B and C temperature though small (Sl. No. 1,3 and 5) , also decrease in % IE in serial No. 2,7 and 8

Table-6: Comparison of the Actual with the predicted result for corrosion inhibition of mild steel in 2 M HCl using Karanja seed oil

Sl. No.	Inhibitor concentration (% v/v)	Time (min)	Temp. (^{o}C)	Inhibition Efficiency (%)	Predicted	Residuals
1	2	1	27	55.26	58.01	2.75
2	10	4	27	94.72	91.97	-2.75
3	10	4	30	74.51	77.01	2.5
4	10	1	30	66.75	65	-1.75
5	10	1	27	86.83	88.83	2
6	2	4	30	43.03	45.28	2.25
7	2	1	30	35.17	32.17	-3
8	2	4	27	63.21	61.21	-2

3.2 The Pareto Chart And Normal Probability Chart

The chief properties & their connections are realistically accessible by Pareto chart in Fig. 1. The erect stroke in Fig. 1 specifies least statistically significant result scale for a 95 % sureness level. Its detected for 95 % confidence level & 8 freedom grades, the t value is 2.82. Altogether values awarding an absolute value greater than 2.82, which are situated right of erect line, are significant. Analyzing the Pareto chart, it can be seen that the contact of concentration had the greatest effect on the corrosion inhibition of mild steel by pongamia oil. The second interaction affecting the errosion inhibition was the temperature followed by time and finally by (conc. &temp).

Usual probability plot has been be detached into two regions, the factors above 50% region are indicated positive coefficients effect and below 50% region indicated as negative coefficients effect. All these issues & connections signified as a circle are not significant and the effects shown as a square is significant. The main factors (A, B, and BC) are away from the straight line and are therefore considered to be real. A, B and BC on the right has a positive effect for corrosion inhibition. From fig. 12 the concentration (A) has largest effect because its point lies furthest after the line. The 2nd vital factor is duration (B) which is additional significant than C (temperature).

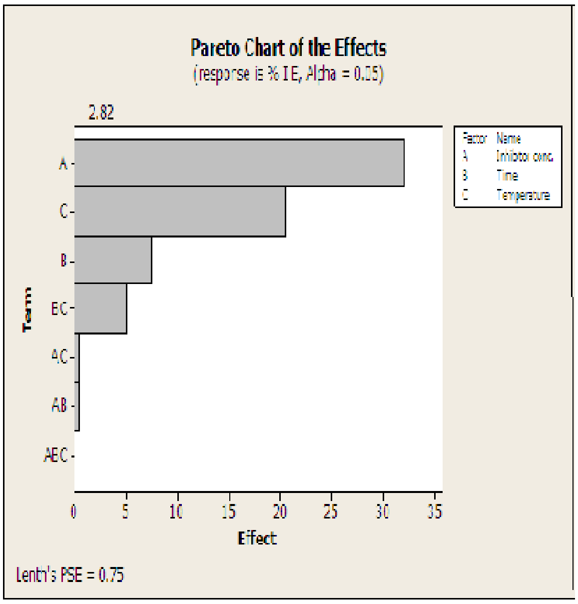

Figure-1
Pareto chart of the standardized effects for the corrosion inhibition of mild steel

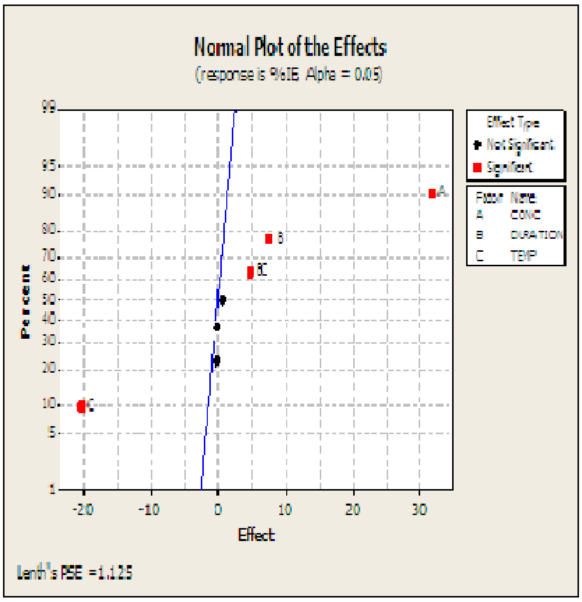

Figure2
Normal probability plot for the effects of input variables for each substance studied

3.3 The Main Effects And The Interaction Effects

Major variations that happen in the reaction (%IE) once the heights of every single of the key features are altered from small to great level are showed in diagram. 3. The statistical meaning of a issue is straight correlated to the size of erect line. Similarly it has noted that the higher the erect line, the more the variation in the reaction when altering the main factor beginning level -1 to level +1. Therefore, from Figure.3, it can be decided that the conc., and time have +ve effect on corrosion inhibition by pongamia oil, once 2 features are altered from small to great & the contradictory is true for the temperature. From the length of the erect lines, it is observed that the duration had the bottom effect on corrosion inhibition.

An interaction is real once the alteration in the result from small to great heights of feature is relied on the level of a 2nd factor, i.e., when the lines do not run parallel. Fig 4 shows the collaboration plots % IE of mild steel using pongamia oil. It can be understood that here is a strong interactional result among adsorbent quantity and temperature. Alternatively, the collaboration among the period and temperature was not effective.

As can be seen from Fig. 4, when the temperature is increased from 27 to 30°C, the % IE is decreased 74 to 35.17 at an inhibitor dosage of 10 (%v/v) and 4 days duration. Therefore, its decided that the result of low temperature is more distinct at high concentration of inhibitor and at long duration[4].

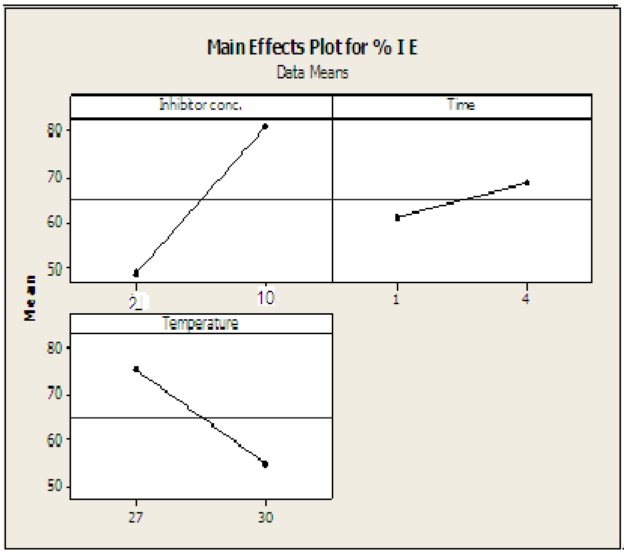

Figure-3
Main effects plot for % Effects Plot for % IE using pongamia oil

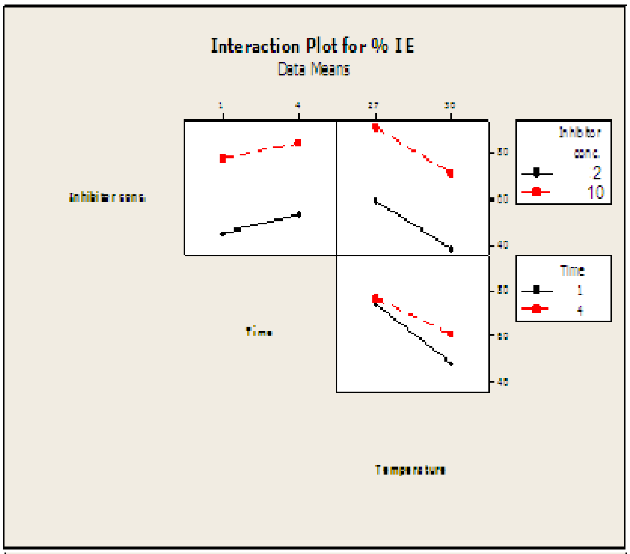

Figure- 4
Interaction effect plot for % IE using pongamia oil

4.0 Conclusions

A preliminary investigation is done with the factorial design of experiments for corrosion inhibition of mild steel using pongamia oil under corrosive media. The effects of 3 factors; inhibitor concentration (2-10 v/v%), time (1-4 days), and temperature (27-30 °C) on % of inhibition remained known. The statistical examination for each case established that the equation (Eq. (8)) gave a reasonably good fit. According to the significance effect obtained in variance analysis, the inhibitor concentration(10 v/v%), with 4 days duration and low temperature of 27 °C was found to have a significant effect on the errosion inhibition of MS in 2M HCl

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