
FORMULATION AND OPTIMIZATION OF THE ORGANIC FILM -FORMING INHIBITOR FOR CONTROL OF CORROSION IN OIL FIELD USING DESIGN EXPERT SOFTWARE.

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Abstract: This involved the formulation of organic film forming inhibitor that is intended to be used in control of corrosion in petroleum oil field environment. Preliminary investigation was carried out as a means of range finding by manually varying the concentrations of the components of the inhibitor. The obtained percentage concentrations ranges were optimized using design expert version 7.0. The experimental design method used was Mixture Design Techniques (D-Optimal method). This was used to determine the optimum concentration of the components in the inhibitor formulation. The combined therapy of formulation D gave performance with efficiency of 99.92% followed by formulation C with efficiency of 99.70%. The other formulations A and B also gave efficiency of 99.685 and 99.69 respectively with the different values of octadecylamine and imidazoline concentration. The best optimum performance is that of formulation D containing 18.41% octadecylamine, 24.15% of imidazoline, 1.27% surfactant, 0.4% demulsifier, 1.4% inorganic synergy 1.24% co-solvent and 52.3 solvent.

Keywords: Formulation, Organic Film Forming, Mixture Design Techniques, Combined Therapy, Optimization.

INTRODUCTION

Corrosion can be defined as the destructive attack of a metal by chemical or by electrochemical reaction with its environment (Redmond, 2008). The isolation of a metal from corrosive agents is the most effective way to prevent electrochemical corrosion. A corrosion inhibitor is a substance which, when added to an environment, in small quantity decreases the rate of attack by the environment on a metal (Wikipedia 2009). Some inhibitors function by being adsorbed on the corroding metal surface, creating a barrier which isolates the metal from the corrosive agents (Umoren *et al*, 2011). One of the suitable means for achieving this purpose is the use of corrosion inhibitors. Inhibitors can be organic, inorganic, polymeric, simple or complex formulations (HTS Consultants, 2004). The effectiveness of the inhibition process depends on such factors as the inhibitor structure and concentration, the nature of the metal surface including the population of potential adsorption sites, temperature and composition of the corrosive environment. (Umoren *et al*, 2011). Imidazolines are good inhibitors, temperature stable and have neutralizing ability (Vladimir *et al*, 1998). However, it has been found to aggravate localized corrosion by creating a small number of major anodes that focused on a small area of the material surface (Okafor *et al*, 2009). In order to address this, there is need to optimise the inhibitor formulation and combining it with other materials that may reduce the localized corrosion effect. The optimization of inhibitor mixing process conditions is one of the most critical stages in the development of an efficient and economic inhibitor mixture. Statistical methodologies involve use of mathematical models for designing inhibition processes and analyzing the process results. Mixture design technique using D-optimal method is a powerful mathematical model with a collection of statistical techniques where in, interactions

	- Used in equal proportion	
Co-solvent	<p>Fatty amide R = C₁₈</p> $\begin{array}{c} \text{O} \\ \parallel \\ \text{R}-\text{C} \\ \\ \text{NH}_2 \\ \text{Amide} \end{array}$	<p>To achieve a clear liquid product with acceptable stability Improves the inhibition effect because it is a mild inhibitor</p>
Solvent	<p>Ethylene Glycol HO - CH₂ - CH₂ - OH Used with water in equal proportion</p>	<p>To reduce product viscosity to a level that will enable product to be pumped under likely conditions of temperature to be encountered in use.</p>

Formulation of the Inhibitor of Different Concentrations.

Preliminary investigation was carried out to determine the range at which the various components of the inhibitor can perform without causing compatibility problem. The choice of the components were made based on the ability of the components forming hydrogen bond between themselves for proper mixing. The primary inhibitor concentrations were varied between 0 and 40 % and the other components were also varied. The range shown in table 2.2 was established.

Table2.2: Ingredients, Components and Overall Percentage Concentration of the Inhibitors.

Ingredients	Components	Overall Percentage concentration
Primary Inhibitor Base	i) Octadecylamine	
	ii) Imidazoline	
	Total volume	10 - 40
Surfactant	i) Ethanol amine	
	ii) Diglycol amine	
	Total volume	2
Demulsifier	Silicone	1
Inorganic synergies	i) (NH ₄) ₂ SO ₃	
	ii) (NH ₄) ₂ S ₂ O ₃	
	Total volume	6
Co-solvent	Octadecylamide	8
Solvent	i Ethylene Glycol	
	ii Water	
	Total volume	73- 43
	OVERALL TOTAL	100%

The overall percentage concentration in table 2.2 was used as the range for experimental design to enable the optimisation of the inhibitor formulation.

Experimental Design and Statistical Analysis

Design expert software version 7.0 (Stat-Ease, 2005) was used to design and optimize the concentration of each component of the inhibitor in the formulation simultaneously using the overall percentage concentration in table 2.2. The Mixture Design Techniques (D-Optimal method) was used to determine the optimum concentration of the components in the inhibitor formulation. The components were varied based on their percentage concentrations as

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independent variables and the influence of these components on inhibitor efficiency (Y_1) as the dependent variable. This software was used for regression and graphical analysis of the data obtained. The goodness of fit of the model was evaluated by the coefficient determination (R^2) and the analysis of variances (ANOVA). The optimum values of the variables were obtained by graphical and numerical analysis using the Design-Expert program based on the criterion of desirability.

Formulations using design expert version 7.0 were carried out and the Design Constraints was; $x_1+x_2+x_3+x_4+x_5+x_6+x_7 = 100.00$.(2.1)

The seven independent variables and their concentrations at different coded and actual levels of the variables employed in the design matrix are shown in table 2.3. The components were varied based on their percentage concentrations as independent variables and the influence of these components on inhibitor efficiency (Y_1) as the dependent variable were well studied and shown in Tables 2.3 and 3.3.

Table 2.3: Design Summary

Study Type		Initial Design			Design Model		Runs	Blocks	
Mixture		D-optimal, Coordinate Exchange			Quadratic		38	No Blocks	
Comp.	Comp. Name	Units	Type	Low Actual	High Actual	Low Coded	High Coded	Mean	Std. Dev.
X ₁	Octadecylamine	%	Mixture	10.00	40.00	0.00	1.00	17.34	9.53
X ₂	Imidazoline	%	Mixture	10.00	40.00	0.00	1.00	18.49	9.69
X ₃	Surfactant	%	Mixture	0.00	2.00	0.00	0.07	0.60	0.81
X ₄	Demulsifier	%	Mixture	0.00	1.00	v0.00	0.03	0.37	0.45
X ₅	Inorganic synerg	%	Mixture	0.00	6.00	0.00	0.20	2.11	2.43
X ₆	Co-solvent	%	Mixture	0.00	8.00	0.00	0.27	2.78	3.17
X ₇	Solvent	%	Mixture	50.00	80.00	0.00	1.00	58.31	9.54
L_Pseudo Coding			Total = 100.00						
Resp.	Name	Units	Obs	Analy.	Min.	Max.	Mean	Ratio	Tran.
Y1	R1	%	38	Polynomial	90.00	110.00	98.58	1.22	None
Std. Dev.	Model								
5.48	Quadratic								

The regression and graphical analysis of the data was obtained. Thirty eight experimental run was initially suggested by the simulator which after optimization thirty experimental runs were obtained. The aliased part of the model was expunged at the analysis of variance to obtain the final polynomial equation 3.1. The generated final equation for the formulation of the inhibitor compositions was used to carryout the mixing.

RESULT AND DISCUSSION

Experimental Design Result

The percentage concentration of the inhibitor components were optimized using D- optimal method of Mixture Design Techniques. The results obtained from 38 experimental runs and the predicted data from the model based on the experimental data were summarized in Table 3.1. Data were analyzed using Design Expert 7.0 software to yield analysis of variance (ANOVA), regression coefficients and regression equations. P-values <0.01 were regarded as significant and P-values <0.005 as very significant. The standard error of design is 0.35, the model is quadratic. The polynomial final equation in terms of actual components of the quadratic model was 3.1:

$$Y_1 = +1.56X_1 + 1.17X_2 + 819.40X_3 - 294.46X_4 - 18.67X_5 - 10.31X_6 + 1.47X_7 + 0.05X_1X_2 + 8.27X_1X_3 + 3.23X_1X_4 + 0.23X_1X_5 + 0.13X_1X_6 - 0.032X_1X_7 - 8.27X_2X_3 + 3.04X_2X_4 + 0.23X_2X_5 + 0.17X_2X_6 - 0.02X_2X_7 - 10.88X_3X_4 - 7.95X_3X_5 - 8.38X_3X_6 - 8.30X_3X_7 + 3.99X_4X_5 + 3.94X_4X_6 + 2.89X_4X_7 + 0.38X_5X_6 + 0.19X_5X_7 + 0.09X_6X_7 \quad (3.1)$$

The diagnostic case statistic influence report gave a total of 38 experimental run under none transform mode.

Table: 3.1: Diagnostic Case Statistic Influence Report

Response 1 = R1					Transform: None			
Stand-ard Order	Actual Value	Predicted Value	Resid-ual	Lever-age	Internaly Studentized Residual	Externaly Studentized Residual	Influence on Fitted Value DFFITS	Cook's Distance
1	90.00	92.95	-2.95	0.498	-0.809	-0.794	-0.791	0.023
2	103.00	103.61	-0.61	0.952	-0.543	-0.523	*-2.33	0.209
3	94.00	95.20	-1.20	0.496	-0.329	-0.314	-0.312	0.004
4	96.00	96.90	-0.90	0.953	-0.812	-0.797	*-3.58	0.476
5	97.00	98.09	-1.09	0.849	-0.547	-0.527	-1.247	0.060
6	94.00	93.67	0.33	0.960	0.324	0.309	1.519	0.091
7	90.00	91.45	-1.45	0.941	-1.169	-1.193	*-4.79	0.785
8	98.00	98.21	-0.21	0.961	-0.202	-0.192	-0.951	0.036
9	107.0	105.91	1.09	0.868	0.586	0.566	1.450	0.081
10	104.00	103.81	0.19	0.962	0.189	0.179	0.897	0.032
11	98.00	97.76	0.24	0.966	0.249	0.237	1.269	0.064
12	109.00	109.76	-0.76	0.940	-0.604	-0.584	*-2.32	0.206
13	91.00	90.49	0.51	0.907	0.327	0.312	0.973	0.037
14	99.00	101.91	-2.91	0.496	-0.707	-0.782	-0.775	0.022
15	90.00	91.31	-1.31	0.869	-0.704	-0.685	-1.766	0.118
16	99.00	99.19	-0.19	0.965	-0.199	-0.189	-0.991	0.039
17	93.00	96.52	-3.52	0.842	-1.723	-1.949	*-1.449	0.563
18	101.00	101.77	-0.77	0.517	-0.215	-0.204	-0.211	0.002
19	101.00	99.34	1.66	0.913	1.096	1.109	*3.58	0.448
20	94.00	92.89	1.11	0.927	0.798	0.783	*2.78	0.288
21	104.00	96.74	7.26	0.497	1.994	2.438	*2.42	0.140
22	94.00	94.37	-0.37	0.954	-0.336	-0.320	-1.461	0.084
23	102.00	102.30	-0.30	0.979	-0.396	-0.378	*-2.55	0.255
24	107.00	107.54	-0.54	0.498	-0.457	-0.438	-1.865	0.135
25	104.00	99.69	4.31	0.497	1.185	1.212	1.206	0.050
26	93.00	94.24	-1.24	0.778	-0.513	-0.493	-0.924	0.033
27	98.00	96.62	1.38	0.938	1.084	1.095	*4.27	0.639
28	104.00	104.70	-0.70	0.850	-0.352	-0.336	-0.798	0.025
29	96.00	98.77	-2.77	0.293	-0.642	-0.622	-0.401	0.006
30	102.00	101.57	0.43	0.529	0.121	0.115	0.122	0.001
31	110.00	107.00	3.00	0.684	1.038	1.043	1.534	0.083
32	101.00	101.38	-0.38	0.415	-0.096	-0.091	-0.076	0.000
33	100.00	03.89	6.11	0.372	1.502	1.619	1.246	0.048
34	96.00	92.95	3.05	0.498	0.839	0.826	0.822	0.025
35	95.00	99.69	-4.69	0.497	-1.287	-1.337	-1.330	0.059
36	90.00	96.74	-6.74	0.497	-1.850	-2.164	*-2.15	0.121
37	97.00	95.20	1.80	0.496	0.494	0.474	0.417	0.009
38	105.00	101.91	3.09	0.496	0.848	0.835	0.828	0.025

*** Exceeds limits**

Exponging the once that exceeds the limit, it was left with 30 experimental formulations. The difference between the actual and the predicted are not much wide apart except for the standared order number 17,19,21,36 and 38. These are also among those that exceeded the limits.

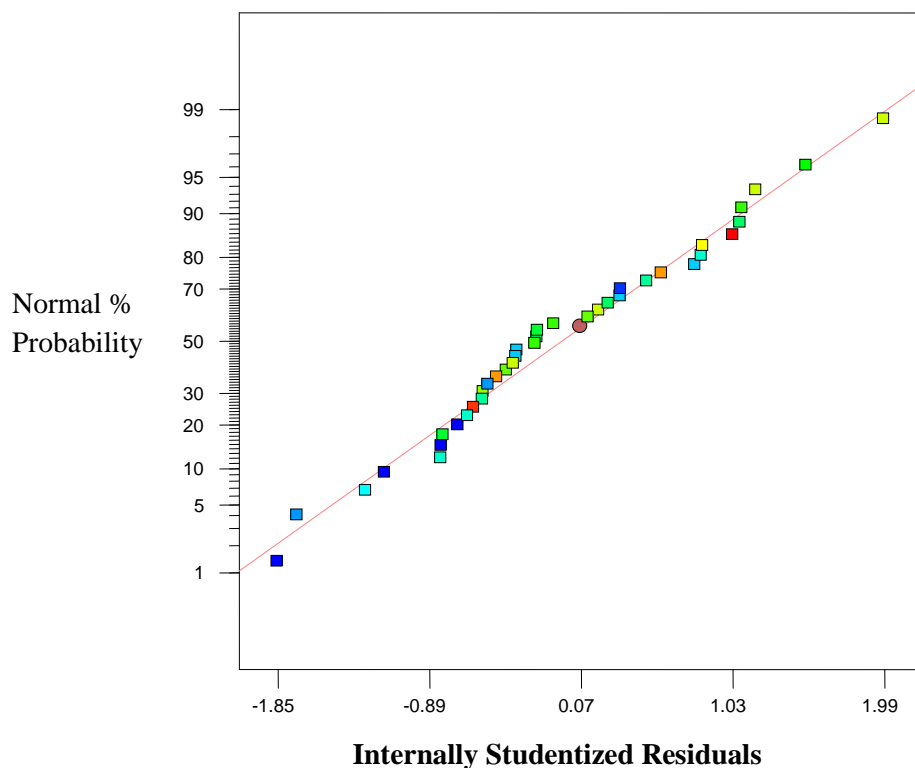


Figure.3.1: Normal Percentage Probabilty Plot against Internally Studentized Residual for Inhibitor Formulation.

The normal percentage probability plot indicates whether the residuals follow a normal distribution, in which case the points were expected to follow a straight line. Expect some moderate scattered points even with normal data. Watch out only for definite patterns like an "S-shaped" curve, which indicates that a transformation of the response may provide a better analysis. The graph of figure 3.1 gave a definite pattern therefore investigation on the residual against predicted plot was carried out.

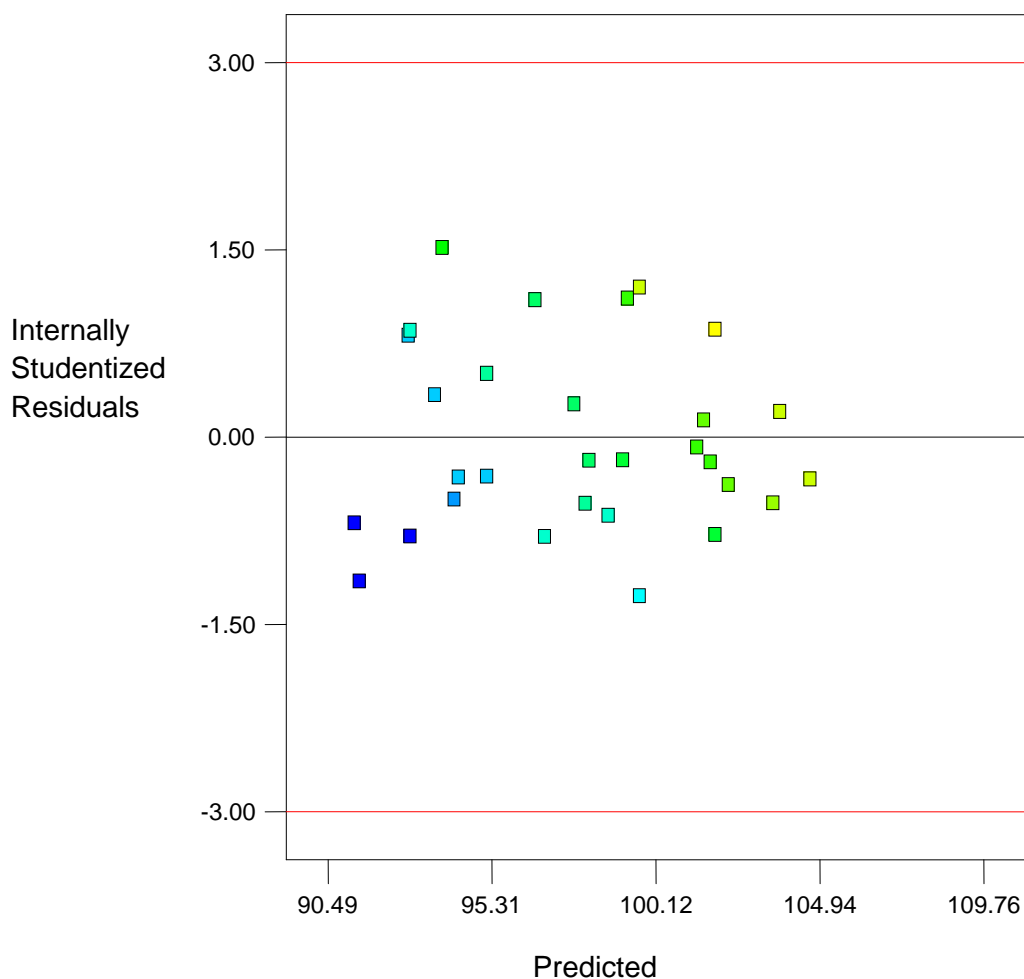


Figure 3.2: Residual against Predicted Plot for Inhibitor Formulation.

This is a plot of the residuals versus the ascending predicted response values. It tests the assumption of constant variance. The plot should be a random scatter (constant range of residuals across the graph.) Expanding variance in this plot indicates the need for a transformation. All the design point seems good because none of them exceeded the line at ± 3.0 line, however the best are those close to the zero point line. The outliers are far from the limit range and are removed from the formulation to give 30 experiments figure 3.2 and 3.3.

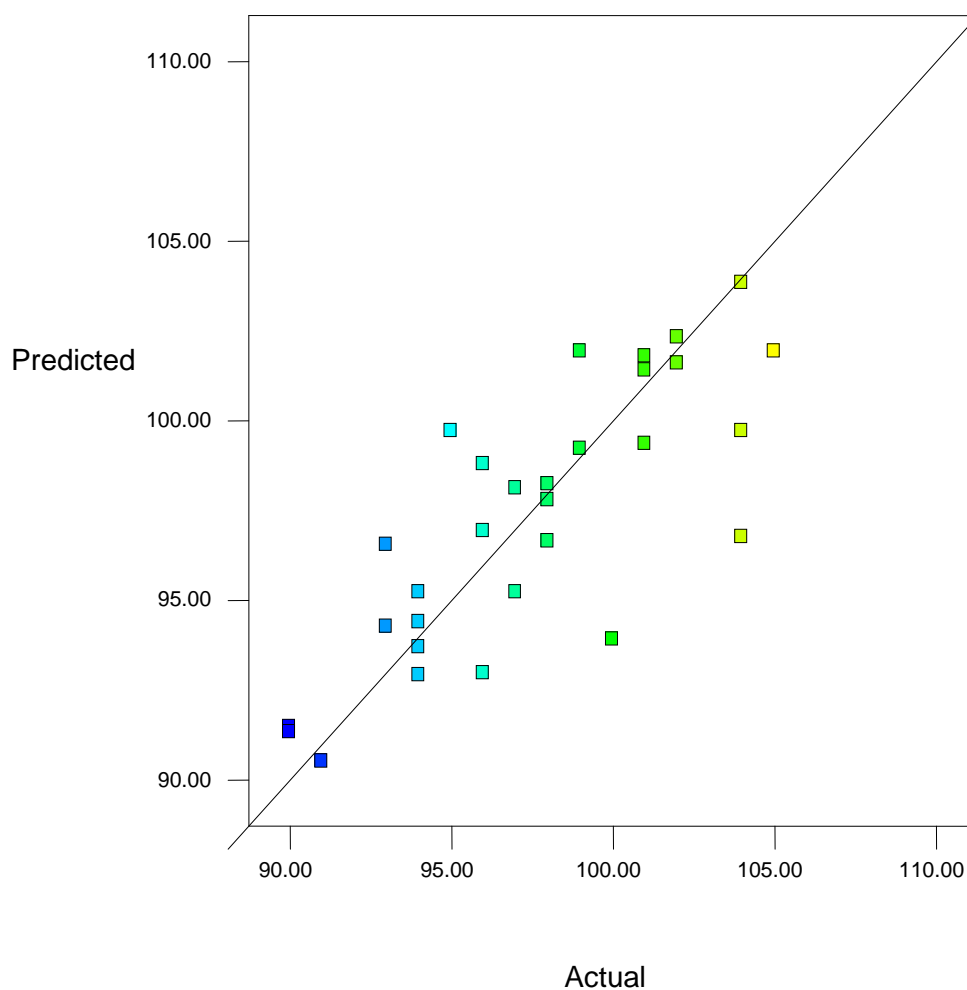


Figure 3.3: Predicted against Actual

A graph of the actual response values versus the predicted response values figure 3.3. It helps to detect a value, or group of values, that are not easily predicted by the model. The data points should be split evenly by the 45 degree line. The plot of figure 3.3 followed the 45 degree line with a few outliers. This leads to the performing of a transformation using the Box Cox plot to improve the fit.

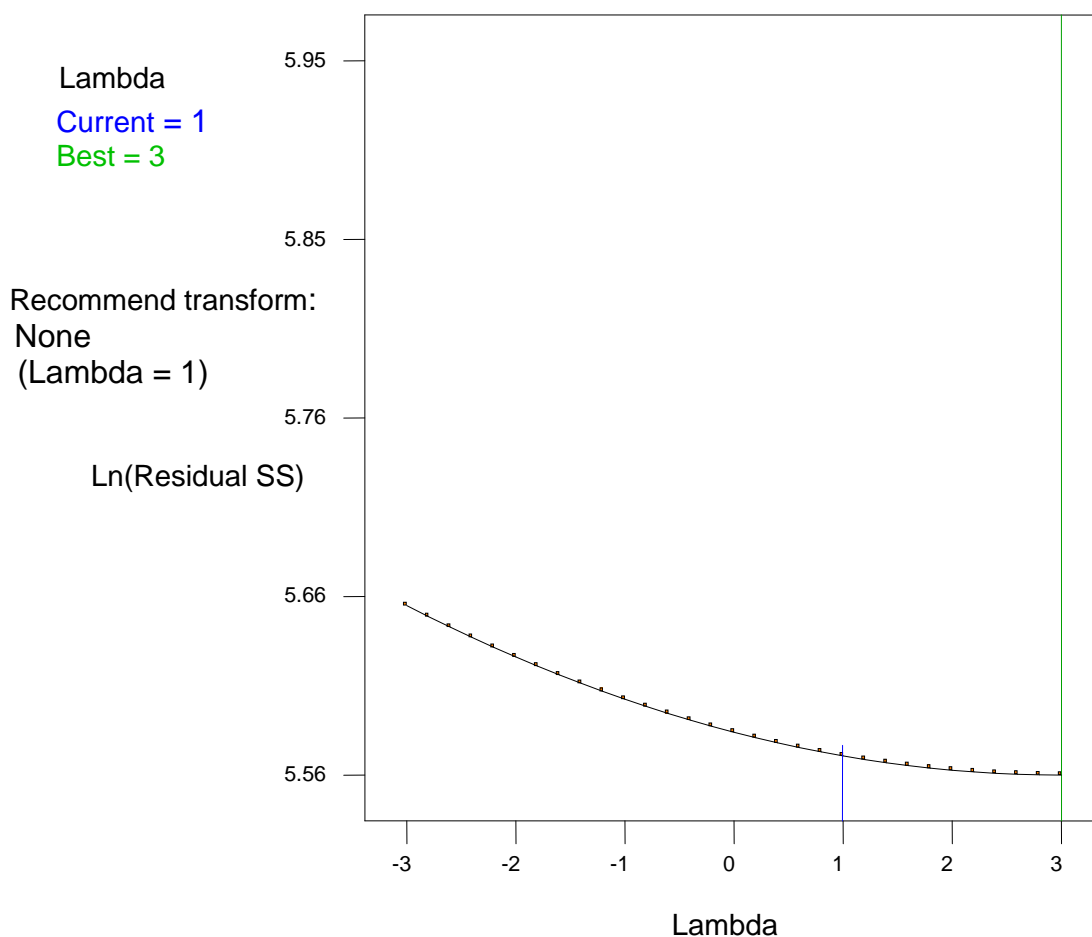


Figure 3.4: Box-Cox Plot for Power Transform

The plot in figure 3.4 (Box Cox) provides a guideline for selecting the correct power law transformation. A recommended transformation is listed, based on the best lambda value, which is found at the minimum point of the curve generated by the natural log of the sum of squares of the residuals. If the 95% confidence interval around this lambda includes 1 then the software does not recommend a specific transformation. The Box Cox plot is not displayed when either the logit or the arcsine square root transformation has been applied. In the figure 3.4 no transformation was recommended because the lambda is equals 1.

The graph of figure 3.5 indicates that equal concentration of octadecylamine (17.4329) and imidazoline (17.4329) will give the highest efficiency response (R_1) of 97% for a two point mixing. The concentrations of other components remain constant as indicated in figure. 3.5.

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◆ **Design Points**

x_1 = octadecylamine
 x_2 = imidazoline

Actual Components

x_3 : surfactant = 0.970
 x_4 : demulsifier = 0.492
 x_5 : inorganic synergy = 2.726
 x_6 : co-solvent = 3.513
 x_7 : solvent = 57.433

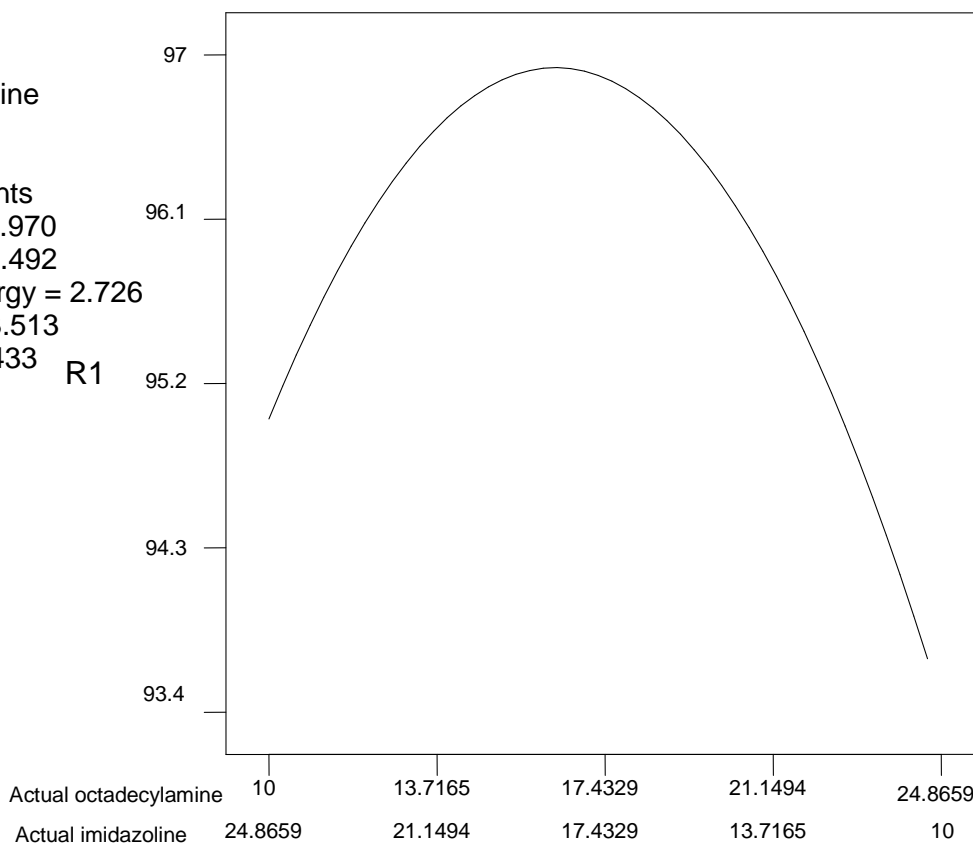


Figure 3.5: Model Graph of Two Points Mixing of the Major Components of the Inhibitor

The overlay plot shows that varying three components x_1 , x_2 , x_3 and leaving the other component constant gave the best performance shown in figure 3.6 with 98.57% efficiency. The variations of the various components are carried out in the table 3.3a to optimise the effect of the components using the new range suggested by the simulator to carry out optimization.

x_1 = octadecylamine

x_2 =: imidazoline

x_3 =: surfactant

Actual Components

x_4 : demulsifier = 0.492

x_5 : inorganic synergy = 2.726

x_6 : co-solvent = 3.513

x_7 : solvent = 57.433

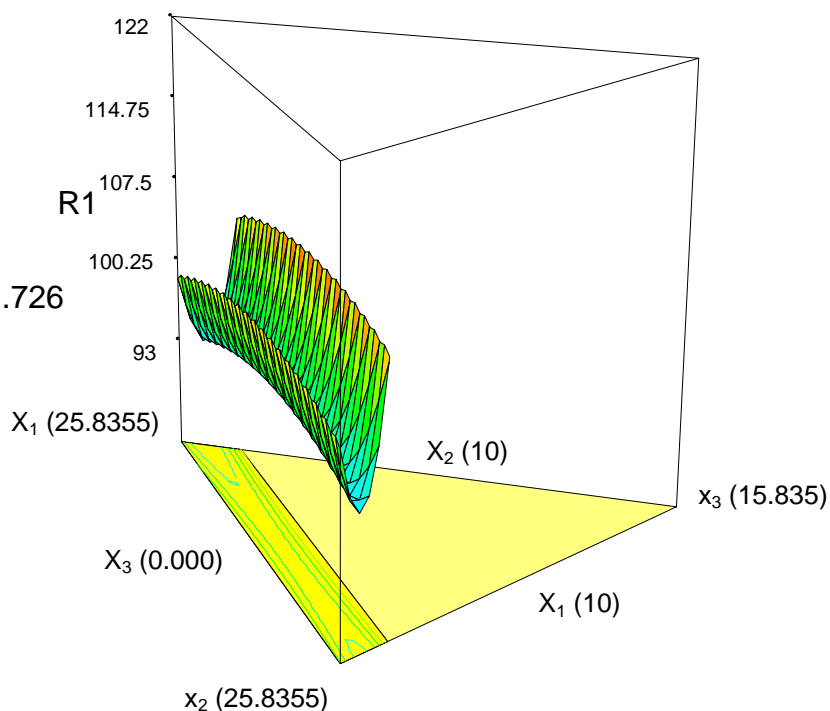


Figure 3.6: Model Graph of Three Dimension Overlay Plot Showing Variation of Three Components of the Inhibitor.

Table: 3.3: OPTIMIZATION

The optimization was carried out with the constraints shown below and the simulator removed outlier. The optimum values of the variables were obtained by graphical and numerical analysis using the Design-Expert program based on the criterion of desirability table 3.3b.

Table: 3.3a: Numerical Optimization result

	Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
X_1	octadecylamine	is in range	10	38.982	1	1	3
X_2	imidazoline	is in range	10	39.0637	1	1	3
X_3	surfactant	is in range	0	2	1	1	3
X_4	demulsifier	is in range	0	1	1	1	3
X_5	inorganic synergy	is in range	0	6	1	1	3
X_6	co-solvent	is in range	0	8	1	1	3
X_7	solvent	is in range	50	79.9981	1	1	3
	R1	is in range	90	100	1	1	3

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Table 3.3b: Compositions of the Components of the Inhibitor with Varying Concentrations as

Solutions Num.	Octadecyl-amine	imidazole	surfactant	demulsifier	inorganic synergist	co-solvent	solvent	R1	Desirability
1	15.876	20.706	0.364	0.261	1.798	0.102	60.893	95.31	1.00
2	17.970	17.535	0.442	0.170	1.887	2.409	59.588	95.82	1.00
3	26.234	11.829	0.088	0.335	3.190	1.953	56.370	98.35	1.00
4	20.103	15.288	0.507	0.441	0.445	0.880	62.336	92.73	1.00
5	21.129	20.534	0.598	0.007	1.212	1.346	55.173	96.76	1.00
6	11.934	19.801	0.913	0.336	1.602	1.217	64.196	94.02	1.00
7	18.225	17.684	0.613	0.375	2.630	1.899	58.574	96.41	1.00
8	18.339	15.803	0.714	0.350	2.005	2.943	59.846	95.03	1.00
9	21.427	18.976	0.461	0.119	1.350	0.667	57.000	96.08	1.00
10	12.813	19.842	0.375	0.535	3.470	3.724	59.241	97.80	1.00
11	16.545	17.631	0.380	0.581	0.766	0.898	63.200	94.11	1.00
12	15.111	19.873	0.809	0.300	0.329	0.628	62.950	92.61	1.00
13	10.008	25.546	0.721	0.448	0.988	2.865	59.424	92.56	1.00
14	12.731	25.446	0.273	0.337	2.672	0.096	58.446	95.59	1.00
15	17.141	16.179	0.774	0.211	2.568	3.220	59.906	95.26	1.00
16	18.530	20.051	0.559	0.573	2.271	1.601	56.415	97.63	1.00
17	21.720	19.508	0.843	0.100	0.689	1.428	55.713	96.13	1.00
18	22.444	21.222	0.490	0.051	0.741	3.098	51.954	99.05	1.00
19	12.960	10.987	1.016	0.190	1.500	6.254	67.094	91.44	1.00
20	16.637	18.245	0.987	0.055	2.637	0.928	60.512	96.12	1.00
21	14.913	23.099	0.952	0.487	2.452	3.066	55.031	97.87	1.00
22	14.569	14.694	0.017	0.240	0.803	3.419	66.256	97.65	1.00
23	10.204	23.246	1.008	0.486	2.686	1.825	60.545	94.11	1.00
24	17.466	20.291	0.138	0.314	1.925	0.215	59.650	97.81	1.00
25	20.333	11.542	0.371	0.089	2.229	3.110	62.326	93.44	1.00
26	24.926	12.744	0.386	0.379	5.160	4.938	51.467	99.45	1.00
27	11.095	27.503	1.274	0.189	3.148	1.539	55.251	97.81	1.00
*28	<u>23.081</u>	<u>21.195</u>	<u>0.924</u>	<u>0.157</u>	<u>0.654</u>	<u>2.380</u>	<u>51.609</u>	<u>99.69</u>	<u>1.00</u>
29	26.749	16.014	1.302	0.724	0.519	1.966	52.727	97.76	1.00
30	19.452	11.857	0.454	0.440	1.679	3.422	62.694	93.80	1.00

Suggested by Design Expert 7.0 after Initial Adjustment.

***Solution number 28 was Selected**

The best performance in the numerical optimization is the solution number 28 with response of 99.69%, followed by solution number 26 with response of 99.45%, therefore solution number 28 was selected to be one of the bases of our formulation called formulation A. Also graphical carried optimisation was out to obtain the overlay plot in fig 3.7, called formulation B.

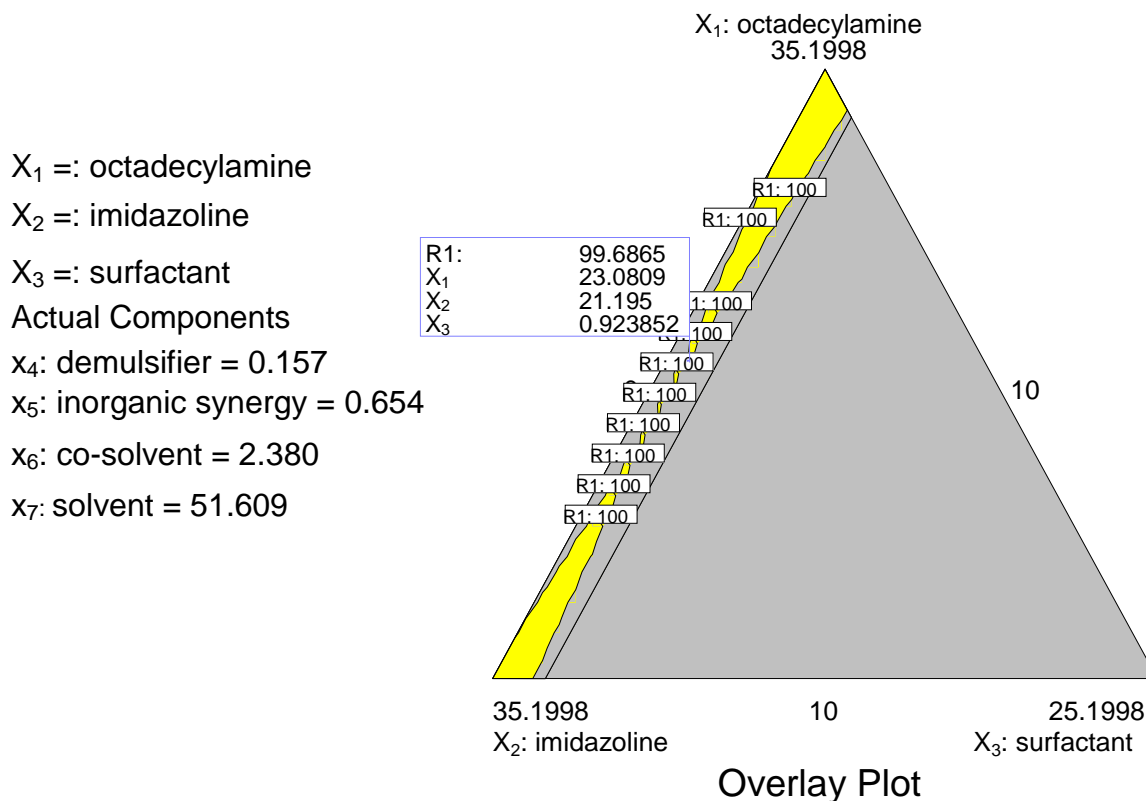


Figure .3.7: Overlay Plot of Graphical Optimization for Varying Components of X_1, X_2 and X_3 .

Point prediction gave a better optimization result in formulation as shown in table 3.4, 3.5 which we called formulation C and D. The prediction of formulation C is 99,7014% while that of formulation D is 99.9275%.

Table 3.4: Formulation C

Component	Name	Level	Low Level	High Level	Std. Dev.	Coding	
X_1	octadecylamine	20.78	10.00	40.00	0.000	Actual	
X_2	imidazoline	22.17	10.00	40.00	0.000	Actual	
X_3	surfactant	0.37	0.000	2.00	0.000	Actual	
X_4	demulsifier	0.36	0.000	1.00	0.000	Actual	
X_5	inorganic synergy	1.4	0.000	6.00	0.000	Actual	
X_6	co-solvent	1.09	0.000	8.00	0.000	Actual	
X_7	solvent	53.78	50.00	80.00	0.000	Actual	
	Total =	100.00					
Response	Prediction	SE Mean	95% CI low	95% CI high	SE Pred	95% PI low	95%PI high
R1	99.7014	3.55	91.80	107.61	6.24	85.79	113.61

Table 3.5: Formulation D

Component	Name	Level	Low Level	High Level	Std. Dev.	Coding
X ₁	octadecylamine	18.41	10.00	40.00	0.000	Actual
X ₂	imidazoline	24.15	10.00	40.00	0.000	Actual
X ₃	surfactant	1.27	0.000	2.00	0.000	Actual
X ₄	demulsifier	0.72	0.000	1.00	0.000	Actual
X ₅	inorganic synergy	1.40	0.000	6.00	0.000	Actual
X ₆	co-solvent	1.74	0.000	8.00	0.000	Actual
X ₇	solvent	52.3	50.00	80.00	0.000	Actual
	Total =	100.00				

Response	Prediction	SE Mean	95% CI low	95% CI high	SE Pred	95% PI low	95%PI high
R1	99.9275	4.23	90.50	109.36	6.65	85.10	114.76

Those that gave better performance from the above simulations are within the range of 15% to 30% of imidazoline and octadecylamine concentration. The best performance was 18.41 octadecylamine and 24.15 imidazoline concentration table 3.5. This is followed by 20.78 octadecylamine and 22.17 imidazoline concentration in table 3.4. The standard error of mean of formulation C is 3.55 compared to that of formulation D which is 4.23 giving a higher error of design. Also the standard error of prediction is higher in formulation D (6.65) compared to that of formulation C (6.24). This indicates that formulation C is more closer to the optimisation constraint than formulation D. However the difference is not much any of the formulation can give high prediction performance as indicated in the table 3.4 and 3.5.

CONCLUSION

The formulation D gave performance with efficiency of 99.92% followed by formulation C with efficiency of 99.70%. The other formulations A and B also gave efficiency of 99.685 and 99.69 respectively with the same values of octadecylamine and imidazoline concentration. The best optimum performance is that of formulation D containing 18.41% octadecylamine, 24.15% of imidazoline, 1.27% surfactant, 0.4% demulsifier, 1.4% inorganic synergy 1.24% co-solvent and 52.3 solvent. The variation of many components at the same time also proved to be the better formulation as compared to that of two components with 97% efficiency and three components with 98.5% efficiency response.

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