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Optimization of product performance of a paint formulation using a mixture experiment

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ABSTRACT *A paint manufacturing company was facing the problem of Vehicle Separation and Settling in one of its prime products. These two abnormalities are, in general, opposing in nature. The manufacturer tried several modifications in the existing recipe for the product but failed to control them. Experimentation was carried out using mixture design, a special type of designed experiment, and quadratic response surface models were fitted for both the responses. Finally,* optimum *formulation was obtained by simultaneously optimizing the two response surface models. During the determination of optimal formulation, different methods were compared. The* optimum *formulation is currently being used for regular manufacturing.*

1 Introduction

In good painting practice, finishing coats are usually applied over an undercoat. The function is to hide the surface and to provide a smooth, uniform foundation for the finishing coat. Primer is a type of undercoat. If the primer fails to do the job expected of it, then the finish will not be satisfactory.

Stoving Primer Surfacer is a widely used primer. It is a mixture of extenders (such as Barrytes, Forcal S, etc), wetting agents (such as Soyalicithin, etc), anti-settling agents (such as Nilset 117, Smaketon Gel, etc), colour pigments, resin, solvent, and additives.

However, in this case, the primer was suffering from the problem of Vehicle Separation and Settling and the manufacturer was receiving a number of complaints from its customers. Separated vehicles (i.e. the liquid portion of the paint, in which pigments and extenders are dispersed) can be dissolved by shaking, but settled pigments and extenders cannot be so dissolved. Moreover, these two abnormalities are conflicting in nature, i.e. if corrective measures are taken to reduce the effect of one, the effect of the other becomes more severe. Several modifications to the existing recipe were tried out to overcome this problem, but none were able to control both the abnormalities simultaneously. Under this circumstance, the manufacturer decided to initiate a study to obtain a primer formulation, which would minimize the occurrence of Vehicle Separation and Settling simultaneously.

2 Background process

The primer is manufactured by mixing different ingredients. The amount of each ingredient is governed by a corresponding recipe. The recipe gives the amount of ingredients in terms of percentage of the total weight. The pigments, extenders and solvents are mixed in a mixer fitted with agitators to produce a paste. The paste is then transferred to the dispersion mill (Bead Mill or High Speed Dispersion Mill) on which further dispersion of the mixture is effected. The degree of dispersion depends upon the character of the end product. The dispersed mixture is then sent to the tanks where more resin, solvents and other additives are added and the shade is adjusted with colour pigments. During this operation, the mixture is continuously stirred using mechanical devices (agitators). One complete production cycle takes about 72 hours of time. It may be noted that 124 kg of input material produces 100 litres of the Primer (specific gravity being 1.24). Henceforth, the ingredients will be termed as *components* and will be expressed in terms of the *proportion* of total weight only.

3 Objectives

The objectives of the study were:

(i) to fit response surfaces for both the responses, namely Vehicle Separation and Settling, and
(ii) to find the optimum levels of the experimental components that would minimize the occurrence of the problem.

4 Approach

Since the main objective is to find the optimum levels of the component proportions, it was decided to carry out a mixture experiment—a special type of designed experiment. Therefore, the following approach was considered to be appropriate:

- carrying out the mixture experiment,
- fitting of response surfaces, and
- optimization of the fitted responses.

For a good discussion on mixture design and the analysis of mixture data, see Cornell (1990).

5 Identification of components

To determine the components responsible for settling and vehicle separation, a brainstorming session was held with concerned technical and R&D personnel and the following components were selected.

TABLE 1. Components with corresponding bounds (in proportion of total weight)

Component	Code	Upper Bound [U]	Lower Bound [L]
Barrytes	x_1	0.175	0.125
Forcal S	x_2	0.050	0.000
Soyalicithin & Nilset 117 [1:1]	x_3	0.006	0.001
Smaketon Gel	x_4	0.020	0.000

(i) *Soyalicithin*: a wetting agent—if increased (decreased), the vehicle separation is increased (decreased), and settling decreased (increased).
(ii) *Forcal S*: an extender—if increased (decreased), vehicle separation is increased (decreased), but there is no effect on settling.
(iii) *Barrytes*: an extender of high Specific Gravity (4.3)—the popular belief was, because of its high specific gravity, this will be more prone to settling.
(iv) *Nilset 117*: an anti-settling agent.
(v) *Smaketon Gel*: reduces settling with no effect on vehicle separation.

It was further decided, from technical consideration, that Soyalicithin and Nilset 117 will be used in a 1:1 ratio.

6 Selection of feasible experimental region

Following the discussion with the technical and R&D personnel, it was decided to investigate the above components within the bounds given in Table 1.

It was further decided to restrict the sum of above four components to 0.1885. Other ingredients, excepting solvents and additives, in the recipe were kept constant. Solvents and additives were suitably adjusted to bring the sum of all components to unity.

7 Construction of the experimental design

Due to the presence of both lower and upper bounds, the feasible mixture region is no longer a simplex, most commonly used mixture design, but will be some polytope (or hyperpolytope) sub-region inside the original unconstrained simplex. Therefore, standard simplex-type designs cannot be used directly. In such cases, some type of computer-generated design is used.

There are several approaches to constructing designs for constrained mixture experiments. A canonical form of a quadratic mixture model, as proposed by Scheffe (1958), namely

$$E(y) = \sum_{i=1}^{4} \beta_i x_i + \sum_{i=1}^{3} \sum_{j=i+1}^{4} \beta_{ij} x_i x_j \tag{1}$$

was considered to be adequate to represent the variability in the responses. To obtain a uniform spread of design points over the feasible mixture region, a computer-generated Distance Based design was used for experimentation. See Myers & Montgomery (1995) for a brief outline of distance-based design methodology. The model (1) has ten parameters that must be estimated. To check the adequacy of the fitted model and to obtain an estimate of the experimental error some additional runs were included in the design. Consequently, a design with 14 runs was initially selected and, afterwards, three runs with high leverage values were repeated to obtain a design with relatively uniform distribution of leverage. Repeat points were also used to check the adequacy of the fitted model through the lack-of-fit test. The G-efficiency of the above design was estimated as 66.30%.

Both the responses, namely Vehicle Separation (y_1) and Settling (y_2) were observed for each of the 17 experimental runs. Primers prepared as per each experimental run were kept in measured cylinders and the responses were observed. Vehicle separation was measured in mm, with no separation (i.e. an observed value of 0 mm) as the most preferred situation, and settling was ranked between 0 to 10, with 10 as the best and 0 as the worst.

The experimental design along with corresponding responses is given in Appendix. The left side panel gives the actual proportions for the mixture components used for each run in the design. The centre panel gives the mixture proportions expressed in terms of pseudocomponents. If x_i denotes the ith original component, then the corresponding pseudo-component X_i is defined as:

$$X_i = \frac{x_i - L_i}{0.1885 - \Sigma_{i=1}^{4} L_i}, \text{ where } L_i = \text{lower bound of the } i\text{th component.}$$

This transformation yields pseudo-components X_i, such that $0 \leqslant X_i \leqslant 1$, $i = 1, 2, 3, 4$ and $\Sigma X_i = 1$, which is the fundamental mixture requirement for obtaining a model of type (1). The right side panel gives the observed values of the responses.

8 Fitting the response surfaces

To reduce the effect of natural multicollinearity or ill-conditioning that is present when the method of least squares is used for estimating the parameters, it was decided to build the mixture response surface models in terms of pseudo-components (see Montgomery & Voth, 1994). The adequacy of the fitted models was judged through various summary statistics, including a lack-of-fit test, adjusted R^2, PRESS (PRediction Error Sum of Squares) and the R^2 prediction statistic (see Myers & Montgomery, 1995, for definitions of the summary statistics). Data were analysed using computer programs written by the author.

Table 2 shows the results of fitting linear and quadratic models sequentially to the data on Vehicle Separation.

TABLE 2. Mixture model building summary statistics for Vehicle Separation (y_1)

Lack-of-fit test

Model	Sum of squares	DF	Mean square	F-value	Prob > F
Linear	350.913	10	35.091	93.58	0.0016
Quadratic	5.497	4	1.374	3.66	0.1574
Pure error	1.125	3	0.375		

Summary statistics of model's fit

Source	Residual DF	Root MSE	R-square	Adjusted R-square	PRESS	R-square prediction
Linear	13	5.20	0.2356	0.0592	547.611	(−)ve
Quadratic	7	0.97	0.9856	0.9671	43.120	0.9064

TABLE 3. Mixture model building summary statistics for settling (y_2)

Lack-of-fit test

Model	Sum of squares	DF	Mean square	F-value	Prob > F
Linear	38.613	10	3.86	4.63	0.1169
Quadratic	0.351	4	0.088	0.11	0.9707
Pure error	2.500	3	0.833		

Summary statistics of model's fit

Source	Residual DF	Root MSE	R-square	Adjusted R-square	PRESS	R-square prediction
Linear	13	1.78	0.2627	0.0926	64.010	(−)ve
Quadratic	7	0.64	0.9489	0.8831	13.733	0.7537

Table 3 shows the results of fitting linear and quadratic models sequentially to the data on Settling.

For both the responses, the quadratic model gave a *higher adjusted R-square value and lower PRESS value*, thereby implying that a quadratic fit would be appropriate, and consequently, a quadratic model was chosen. The quadratic response surface models for Vehicle Separation and Settling, along with other important findings, are given in Tables 4 and 5 respectively.

TABLE 4. Quadratic mixture model for Vehicle Separation (y_1)

Variable	Coefficient	Standard error	t-value
X_1	−475.86	38.38	
X_2	−26.53	6.78	
X_3	2766.25	349.65	
X_4	−349.14	52.76	
X_1X_2	397.92	36.86	10.80
X_1X_3	1302.74	515.71	2.53
X_1X_4	4074.34	270.52	15.06
X_2X_3	−2905.22	389.41	7.46
X_2X_4	651.36	95.94	6.79
X_3X_4	−2717.51	373.61	7.27
R-squared :	0.9856	Adjusted R-squared :	0.9671
PRESS :	43.120	R-squared prediction :	0.9064

ANOVA table of the fitted model

SOV	DF	SS	MS	F	Prob > F
Model	9	453.937	50.437	53.32	0.000
Linear	3	108.521	36.174	38.24	0.000
Quadratic	6	345.416	57.569	60.86	0.000
Residual	7	6.622	0.946		
Lack of fit	4	5.497	1.374	3.66	0.157
Pure error	3	1.125	0.375		
Total	16	460.559			

TABLE 5. Quadratic mixture model for settling (y_2)

Variable	Coefficient	Standard error	t-value
X_1	−179.57	25.18	
X_2	−2.55	4.45	
X_3	1395.32	229.43	
X_4	−156.94	34.62	
X_1X_2	115.29	24.18	4.77
X_1X_3	788.12	338.39	2.33
X_1X_4	1289.57	117.50	7.27
X_2X_3	−1588.66	255.52	6.22
X_2X_4	270.14	62.96	4.29
X_3X_4	−1147.71	245.15	4.68
R-squared : 0.9489		Adjusted R-squared : 0.8831	
PRESS : 13.733		R-squared prediction : 0.7537	

ANOVA table of the fitted model

SOV	DF	SS	MS	F	Prob > F
Model	9	52.914	5.879	14.43	0.001
Linear	3	14.652	4.884	11.99	0.004
Quadratic	6	38.262	6.377	15.66	0.001
Residual	7	2.851	0.407		
Lack of fit	4	0.351	0.088	0.11	0.971
Pure error	3	2.500	0.833		
Total	16	55.765			

It is interesting to note from the model for settling, that the variable x_3 (i.e. Soyalicithin and Nilset 117 combination) has a greater contribution towards settling than x_1 (i.e. Barrytes). This goes against the popular belief that Barrytes is more prone to settling.

The following diagnostic statistics (see Myers & Montgomery, 1995 for definitions) were also calculated for each experimental run, corresponding to both the obtained models:

(i) Studentized Residual,
(ii) Cook's Distance,
(iii) *R* Student,
(iv) *H* Diagonals, and
(v) PRESS Residuals.

Different Diagnostic Statistic values corresponding to the models obtained for Vehicle Separation and Settling are given in Tables 6 and 7 respectively. It is evident from the values of different diagnostics, corresponding to both the models, that, in general, there is not much wrong with the data set. Analyses of Studentized Residuals for both models are given in Figs 1 and 2. Studentized residuals were used because the points in mixture design can have substantial differences in their leverage values and studentized residuals account for leverage through the term $(1 - h_{ii})$ that appears in its denominator. Both the normal probability plots, generated by SPSS for Windows (1997), are satisfactory and revealed that there is no apparent problem with normality. However, the plots of Studentized residuals

TABLE 6. Values of different diagnostic statistics corresponding to the model representing variations in Vehicle Separation

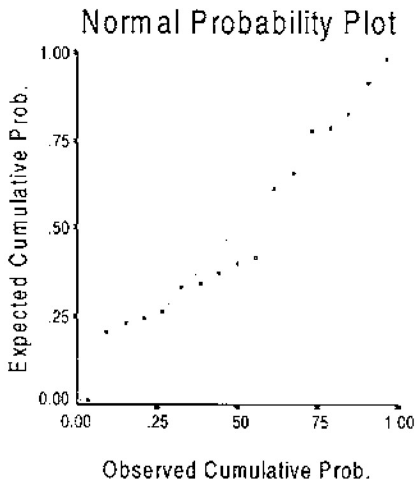
Expt No.	*Y* observed	*Y* predicted	Residual	Studentized residual	Cook's distance	*R* student	Hat(H) diagonal	PRESS residual
1	13.50	14.0000	−0.5000	−0.7270	0.0529	−0.7000	0.5000	−1.0000
2	12.00	13.1712	−1.1712	−2.2309	1.2105	−3.8418	0.7086	−4.0198
3	7.50	6.5841	0.9159	1.3462	0.1891	1.4478	0.5107	1.8718
4	16.50	14.9188	1.5812	2.0692	0.2654	3.0741	0.3827	2.5615
5	10.00	10.5570	−0.5570	−0.7729	0.0491	−0.7482	0.4510	−1.0146
6	8.50	8.6930	−0.1930	−0.4716	0.1034	−0.4437	0.8230	−1.0901
7	15.50	15.7172	−0.2127	−0.4378	0.0576	−0.4110	0.7504	−0.8522
8	21.50	20.9667	0.5333	0.7612	0.0538	0.7359	0.4812	1.0280
9	19.00	19.1664	−0.1664	−0.2497	0.0071	−0.2323	0.5309	−0.3547
10	11.50	11.9766	−0.4766	−0.8628	0.1564	−0.8450	0.6775	−1.4778
11	23.00	22.8891	0.1109	0.2497	0.0237	0.2323	0.7915	0.5320
12	7.50	7.7500	−0.2500	−0.3635	0.0132	−0.3398	0.5000	−0.5000
13	6.00	6.1925	−0.1925	−0.2869	0.0091	−0.2672	0.5240	−0.4045
14	10.00	9.7052	0.2948	0.9024	0.6406	0.8888	0.8872	2.6134
15	14.50	14.0000	0.5000	0.7270	0.0529	0.7000	0.5000	1.0000
16	20.50	20.9667	−0.4667	−0.6662	0.0412	−0.6374	0.4812	−0.8997
17	8.00	7.7500	0.2500	0.3635	0.0132	0.3398	0.5000	0.5000

TABLE 7. Values of different diagnostic statistics corresponding to the model representing variations in Settling

Expt No.	*Y* observed	*Y* predicted	Residual	Studentized residual	Cook's distance	*R* student	Hat(H) diagonal	PRESS residual
1	8.00	7.5000	0.5000	1.1080	0.1228	1.1296	0.5000	1.0000
2	10.00	9.7978	0.2022	0.5869	0.0838	0.5573	0.7086	0.6940
3	5.00	5.0613	−0.0613	−0.1373	0.0020	−0.1273	0.5107	−0.1252
4	9.00	9.2868	−0.2868	−0.5719	0.0203	−0.5423	0.3827	−0.4646
5	6.00	6.2232	−0.2232	−0.4720	0.0183	−0.4441	0.4510	−0.4065
6	4.00	3.9974	0.0026	0.0096	0.0000	0.0089	0.8230	0.0145
7	8.00	7.7539	0.2461	0.7717	0.1790	0.7470	0.7504	0.9858
8	8.00	8.0423	−0.0423	−0.0921	0.0008	−0.0853	0.4812	−0.0816
9	8.00	7.7883	0.2117	0.4844	0.0266	0.4562	0.5309	0.4514
10	7.00	6.8307	0.1693	0.4671	0.0458	0.4394	0.6775	0.5249
11	9.00	9.1412	−0.1412	−0.4844	0.0891	−0.4562	0.7915	−0.6771
12	4.00	5.0000	−1.0000	−2.2159	0.4910	−3.7549	0.5000	−2.0000
13	4.00	3.9273	0.0727	0.1652	0.0030	0.1533	0.5240	0.1528
14	6.00	6.1075	−0.1075	−0.5015	0.1979	−0.4729	0.8872	−0.9531
15	7.00	7.5000	−0.5000	−1.1080	0.1228	−1.1296	0.5000	−1.0000
16	8.00	8.0423	−0.0423	−0.0921	0.0008	−0.0853	0.4812	−0.0816
17	6.00	5.0000	1.0000	2.2159	0.4910	3.7549	0.5000	2.0000

versus predicted responses indicated that variances of the responses do not depend on the mean level of y and can be considered as constant.

Thus, it was concluded, based on all the above findings, that the quadratic model for the responses were adequate to describe the variability in the corresponding response surfaces.



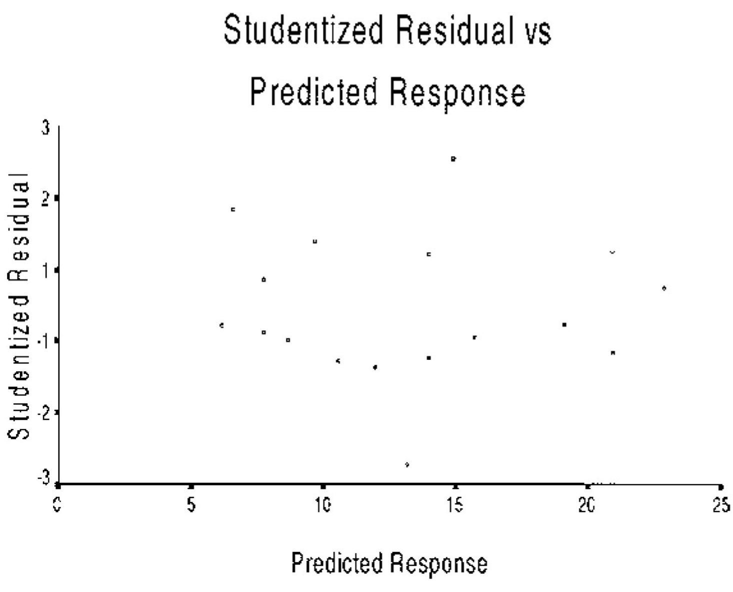


FIG. 1. Analysis of studentized residuals [Response: Vehicle Separation].

The fitted response surface models were:

For Vehicle Separation

$$y_1 = -475.86X_1 - 26.53X_2 + 2766.25X_3 - 349.14X_4 + 397.92X_1X_2 + 1302.74X_1X_3 + 4074.34X_1X_4 - 2905.22X_2X_3 + 651.36X_2X_4 - 2717.51X_3X_4$$

For Settling

$$y_2 = -179.57X_1 - 2.55X_2 + 1395.32X_3 - 156.94X_4 + 115.29X_1X_2 + 788.12X_1X_3 + 1289.57X_1X_4 - 1588.66X_2X_3 + 270.14X_2X_4 - 1147.71X_3X_4$$

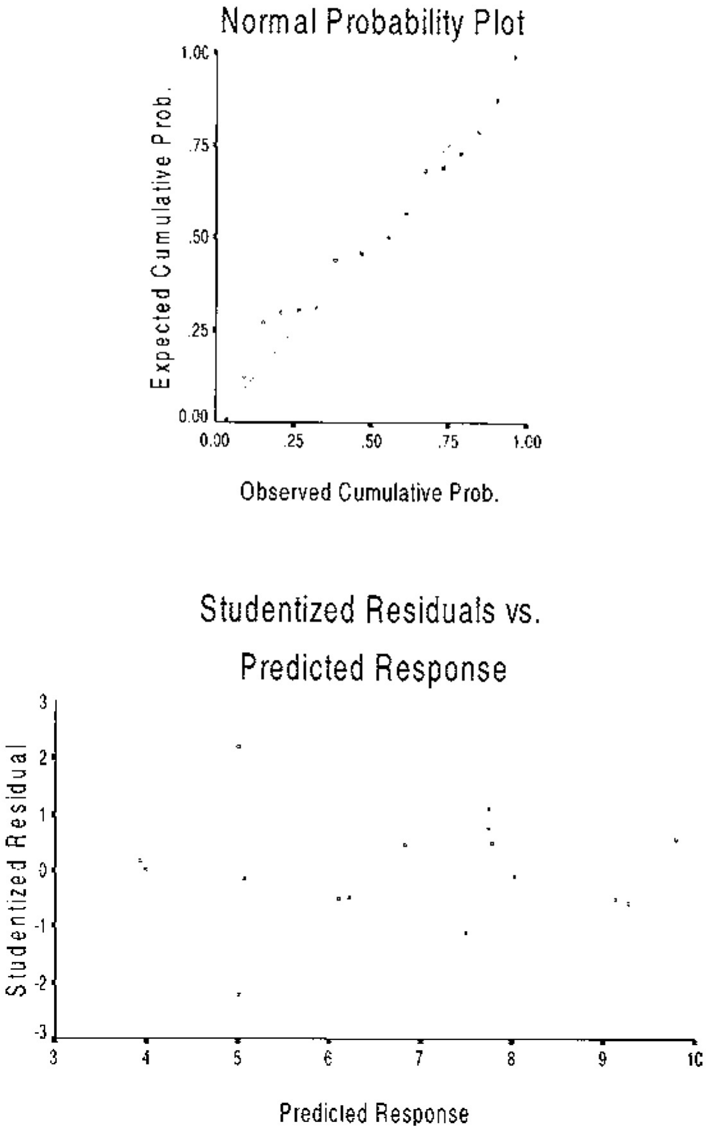


FIG. 2. Analysis of studentized residuals [Response: Settling].

9 Optimizing the primer formulation

The main objective of the study was to find the optimum level of the components x_1, x_2, x_3 and x_4, which would take care of both the abnormalities simultaneously or, in other words, would optimize both the responses simultaneously. Such a situation is widely known as optimization of dual response systems (DRS). Since there were several methods for solving such problem, it was decided to use different available approaches and accept the solution set that corresponded to the minimum cost of production. The following approaches were considered:

(i) non-linear programming approach,
(ii) loss function approach,

(iii) distance function approach, and
(iv) desirability function approach.

9.1 Non-linear programming approach

Del Castillo & Montgomery (1993) used a non-linear programming algorithm to solve such a system. In a DRS problem, the objective is to optimize a primary fitted response surface Y_p subject to the requirement constraint on the secondary response surface Y_s. Here, both the response surface models are assumed to be second-order polynomials. The optimum levels were obtained in terms of the pseudo-components $[X_i]$ and these values were transformed to corresponding actual components using the transformation give below:

$$x_i = [0.1885 - \Sigma L_i] \times X_i + L_i$$

So, to start with, vehicle separation was taken as the primary response, and the problem was formulated as:

$$\text{minimize } y_1,$$

subject to

$$y_2 \leqslant 10,$$

$$y_2 \geqslant 9,$$

$$y_1 \geqslant 0,$$

$$\Sigma X_i = 1.0$$

and X_is $[i = 1, 2, 3, 4]$ were allowed to vary within the experimental region bounded by corresponding lower and upper bounds.

Afterwards, settling was taken as the primary response and the problem was formulated as:

$$\text{maximize } y_2,$$

subject to

$$y_2 \leqslant 10,$$

$$y_1 \geqslant 0,$$

$$y_1 \leqslant 1,$$

$$\Sigma X_i = 1.0$$

and X_is $[i = 1, 2, 3, 4]$ were varied within the experimental region bounded by corresponding lower and upper bounds.

In the above two mathematical formulations y_1 and y_2 represent the models for Vehicle Separation and Settling respectively, in terms of the pseudo-components. These two mathematical programming problems were solved using the SOLVER routine of MS EXCEL (1997) package, which uses a Generalized Reduced Gradient non-linear optimization method. SOLVER reported the same solution for both models. The solution obtained, after reverting to actual components, was:

$$x_1 = 0.1713 \qquad x_2 = 0.0096$$

$$x_3 = 0.0041 \qquad x_4 = 0.0035$$

9.2 Loss function approach

Next, the problem was reformulated using the quadratic loss functions promoted by Taguchi *et al.* (1989). Let Y be the quality characteristic of a product and T its target value, then according to the quadratic loss function, the quality loss is given by

$$\text{Loss}(Y) = k(Y - T)^2$$

where k is the quality loss coefficient. Neon Artiles-Leon (1996–97), defined k as

$$k = (2/(USL - LSL))^2$$

so that loss becomes *zero* at T and *unity* at USL or LSL. As these loss functions are dimensionless, loss functions corresponding to the two quality characteristics (i.e. responses) were added to obtain the total loss as

$$\text{Total loss} = 4\Sigma\{(Y_i - T_i)/(USL_i - LSL_i)\}^2$$

Since the best value for Vehicle Separation and Settling were 0 and 10 respectively, during total loss calculation, the following values of T, USL and LSL were assumed (see Table 8). Here, basic intention was to fix the target sufficiently close to the corresponding best values.

TABLE 8. Target, *LSL* and *USL* values for the responses

Response	Target	*LSL*	*USL*
Vehicle Separation	0.5	0	1.0
Settling	9.5	9.0	10.0

So the problem was then formulated as

$$\text{minimize } \textit{total loss}$$

subject to

$$\Sigma X_i = 1.0$$

and X_is $[i = 1, 2, 3, 4]$ were varied within the experimental region bounded by respective upper and lower bounds.

The formulation obtained above was again solved using the SOLVER routine of MS EXCEL (1997). The optimal values reported to by Excel, in terms of the actual components, were:

$$x_1 = 0.1729 \qquad x_2 = 0.0078$$

$$x_3 = 0.0041 \qquad x_4 = 0.0037$$

9.3 Distance function approach

Afterwards, the simultaneous optimum of the two responses was obtained using the distance function approach suggested by Khuri & Conlon (1981). This distance function measures the distance of the vector of estimated responses from the estimated *ideal* optimum, i.e. the vector of individual optimas. The optimum level of components was then obtained by minimizing the distance function over the experimental region.

Since one equality constraint [i.e. $\Sigma X_i = 1.0$] exists among the four mixture variables, the number of mathematically independent mixture variables is 3. Hence, any 3 mixture variables can be considered as mathematically independent. Therefore, the first three mixture components were considered independent and the second order polynomial was fitted to the responses with X_1, X_2 and X_3 as controllable variables. The fitted polynomial models, in terms of the pseudo components, were:

For Vehicle Separation

$$y_1 = -349.14 + 3947.61X_1 + 973.97X_2 + 397.88X_3 - 4074.34X_1^2 - 651.36X_2^2$$
$$+ 2717.51X_3^2 - 4327.78X_1X_2 - 54.08X_1X_3 - 839.07X_2X_3$$

For Settling

$$y_2 = -156.94 + 1266.94X_1 + 424.54X_2 + 404.56X_3 - 1289.57X_1^2 - 270.84X_2^2$$
$$+ 1147.71X_3^2 - 1442.42X_1X_2 + 646.26X_1X_3 - 711.10X_2X_3$$

In each iteration, the distance between the *ideal* optimal (ideal optimal was taken as 0 for Vehicle Separation and 10 for Settling) and the estimated responses corresponding to a randomly selected point from the experimental region was calculated. After a sufficiently large number of such iterations, the point that gave the minimum distance was taken as the optimal point. The optimal values, in terms of the actual components, were thus obtained as:

$$x_1 = 0.1693 \qquad x_2 = 0.0118$$
$$x_3 = 0.0041 \qquad x_4 = 0.0033$$

9.4 *Desirability function approach*

The simultaneous optimization of the two responses was obtained by maximizing the overall desirability. Following the method suggested by Del Castillo *et al.* (1996), an everywhere-differentiable desirability function was obtained for both the responses. The geometric mean of the individual desirabilities was taken as the overall desirability. In this formulation, the following minimum, maximum, target values and corresponding desirability values were used.

	Minimum		Target		Maximum	
Response	Value	Desirability	Value	Desirability	Value	Desirability
Vehicle Separation	0.0	0.0	0.5	1.0	2.0	0.0
Settling	8.0	0.0	9.5	1.0	10.0	0.0

The maximization of the overall desirability was again obtained through the MS EXCEL (1997) spreadsheet. The optimal values of the components thus obtained were

$$x_1 = 0.1703 \qquad x_2 = 0.0107$$

$$x_3 = 0.0041 \qquad x_4 = 0.0034$$

This solution resulted in the overall desirability value of 0.8119.

10 Selecting the optimal formulation

With a view to selecting the best among the four product formulations obtained above, it was decided to calculate the cost of these four product formulations. Accordingly, the cost of the five ingredients considered for experimentation, required to manufacture 100 litres of the product, was calculated for each of the four product formulations. Table 9 below gives these cost estimates in addition to other important information.

TABLE 9. Solution sets and corresponding estimated responses and cost/100 litres

Solution set	Component proportions				Estimated response		Total cost of the components per 100 litres of paint
	x_1	x_2	x_3	x_4	Vehicle Separation	Settling	
1	0.1713	0.0096	0.0041	0.0035	0.0215	10.0835	Rs. 129.34
2	0.1729	0.0078	0.0041	0.0037	0.1738	10.0652	Rs. 127.60
3	0.1693	0.0118	0.0041	0.0033	0.0325	9.7553	Rs. 131.61
4	0.1703	0.0107	0.0041	0.0034	0.6270	9.5046	Rs. 130.48

All these four product formulations were presented to the management personnel of the organization and they favoured the second set, as it corresponds to the lowest manufacturing cost (since other costs remain same). The finally accepted components proportion were as follows:

Component	Quality
Barrytes	0.1729
Forcal S	0.0078
Soyalicithin & Nilset 117 [1:1]	0.0041
Smaketon Gel	0.0037

Finally, it was decided to obtain an operating window around the accepted formulation for the ease of control during manufacturing. Consequently, a discussion was held with the technical personnel to obtain the ranges of Vehicle Separation and Settling that would not create problems for the end-user. It was decided, based on the criticality of the problems, that Vehicle Separation in $[0, 4]$ and Settling in $[8, 10]$ can be considered acceptable and this interval of acceptance was used for generating the operating windows.

A simulation program was therefore developed, where each set of optimal component proportions is varied between $\pm 20\%$ of the respective optimal values, subject to the corresponding bounds, i.e. between $\pm$ min $\{20\%$ of $x_i^{\text{opt}}, U_i - x_i^{\text{opt}}, x_i^{\text{opt}} - L_i\}$, where x_i^{opt} is the optimal value of the ith component. During simulation, observations were simulated assuming components follow a normal distribution

TABLE 10. Operating window for the accepted formulation

Component name	Desired value	Operating window		
		Maximum	Minimum	Width
Barrytes	0.1729	0.1749	0.1697	0.0052
Forcal S	0.0078	0.0166	0.0004	0.0162
Soyalicithin & Nilset 117 [1:1]	0.0041	0.0044	0.0037	0.0007
Smaketon Gel	0.0037	0.0050	0.0030	0.0020

with a mean at the respective optimal value and with variability as decided above. The simulation program was run for each solution set and, in each run, 1,00,000 realizations were made. The values of the four component proportions, for which the calculated responses were found to fall within the respective acceptable interval, were recorded. The range of those recorded proportions for each component gave the operating window for that component. The operating window for the accepted formulation is given in Table 10.

11 Implementation

Three batches were prepared at the pilot plant of the manufacturer to verify the performance of the accepted formulation. None of those batches were found to have either the Vehicle Separation or the Settling problem. Accordingly, regular production was carried out. The results were also evaluated at the end-user's premises. The primer was found to be fully satisfactory.

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Appendix

Experimental design (in actual components, x_i and pseudo-components, X_i) and responses (Vehicle Separation and Settling)

Expt. no.	x_1	x_2	x_3	x_4	X_1	X_2	X_3	X_4	y_1 [V.S]	y_2 [Sett.]
1	0.1750	0.0050	0.0035	0.0050	0.8000	0.0800	0.0400	0.0800	13.5	8.0
2	0.1250	0.0500	0.0010	0.0125	0.0000	0.8000	0.0000	0.2000	12.0	10.0
3	0.1250	0.0500	0.0035	0.0100	0.0000	0.8000	0.0400	0.1600	7.5	5.0
4	0.1250	0.0462	0.0010	0.0163	0.0000	0.7400	0.0000	0.2600	16.5	9.0
5	0.1250	0.0450	0.0035	0.0150	0.0000	0.7200	0.0400	0.2400	10.0	6.0
6	0.1250	0.0500	0.0060	0.0075	0.0000	0.8000	0.0800	0.1200	8.5	4.0
7	0.1250	0.0438	0.0060	0.0138	0.0000	0.7000	0.0800	0.2200	15.5	8.0
8	0.1312	0.0500	0.0010	0.0063	0.1000	0.8000	0.0000	0.1000	21.5	8.0
9	0.1300	0.0500	0.0035	0.0050	0.0800	0.8000	0.0400	0.0800	19.0	8.0
10	0.1250	0.0425	0.0010	0.0200	0.0000	0.6800	0.0000	0.3200	11.5	7.0
11	0.1288	0.0500	0.0060	0.0038	0.0600	0.8000	0.0800	0.0600	23.0	9.0
12	0.1325	0.0500	0.0060	0.0000	0.1200	0.8000	0.0800	0.0000	7.5	4.0
13	0.1250	0.0400	0.0035	0.0200	0.0000	0.6400	0.0400	0.3200	6.0	4.0
14	0.1250	0.0375	0.0060	0.0200	0.0000	0.6000	0.0800	0.3200	10.0	6.0
15	0.1750	0.0050	0.0035	0.0050	0.8000	0.0800	0.0400	0.0800	14.5	7.0
16	0.1312	0.0500	0.0010	0.0063	0.1000	0.8000	0.0000	0.1000	20.5	8.0
17	0.1325	0.0500	0.0060	0.0000	0.1200	0.8000	0.0800	0.0000	8.0	6.0