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Overcoming an Inadequate Brake Lining Formulation: *A Tutorial Case Study*

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ABSTRACT

It's not an enviable (or unique) position to be in – you've got a formulation that's performing poorly. The approach of "tweaking" what you already have is logical. In fact, it's probably the fastest and cheapest approach – and most likely the best. So now the big question becomes, "How far can we push this formulation to make a major improvement?" What you're really asking is a statistical question – "What are the extreme vertices that define the limits of my design space?" Extreme vertices that bound your process are those points that are located as far away as possible from other points in the design space. Knowing what these extreme blend points are is important. And knowing the design space's overall centroid is also key, for this spot is the center of your design space. This paper describes how effectively and economically one can design experiments to design new formulations or improve the existing formulation using mixture design.

INTRODUCTION

Friction material is very complicated to formulate because it commonly contains anywhere from 10 to 20 components. Depending on sought-after improvements like enhanced performance, long life, low wear, no braking noise, or reduced cost, the few truly important components that contribute to a solution must be found among the trivial many.

Recently, the author designed a new friction formulation for a brake lining manufacturer to replace an existing mix that was producing unacceptably high friction and wear. Table 1 shows the inadequate base formulation that needed improving. The new formulation's discovery was so effective a breakthrough that specific factor names are omitted throughout this article to protect the proprietary formula. However, the method the author followed is valid for nearly any formulation that you may similarly encounter, so let's look at how it was done.

TABLE 1: Material composition of the previously inadequate base formulation. Twelve components made up the mixture.

MATERIAL	WEIGHT PERCENTAGE
Fiber 1	5
Fiber 2	20
Metal powder	9
Binder	12
Friction modifier	5
Lubricant	5
Six Various Fillers	44
TOTAL	**100**

blends violated total weight percent constraints allowed for the three components.

Extreme vertices are mapped out in Table 4, which contains plus (+) and minus (-) signs indicating, respectively, upper and lower constraint levels. The number of columns to be filled in with + and – signs equals q-1, or two. Blank table cells throughout the table are filled in when the actual constraint numbers are inserted in Table 5.

TABLE 4: Standard experiment setup for constrained mixture design with three factors. Extreme high and low vertices are represented by plus (+) and minus (−) signs.

EXPERIMENT	BINDER (X_1)	FRICTION MODIFIER (X_2)	LUBRICANT (X_3)
1	−	−	
2	+	−	
3	−	+	
4	+	+	
5	−		−
6	+		−
7	−		+
8	+		+
9		−	−
10		+	−
11		−	+
12		+	+

Table 5 shows the constraint numbers substituted for the + and – signs. For example, in experiment 1, X_1 and X_2 are at their lower levels of 13 and 10, respectively, adding up to 23. Because $X_1+X_2+X_3$ must equal 30 at all times, the value for X_3 becomes seven. The remaining experiment values are similarly filled in.

The experiments violating individual or total constraints are shown in red and eliminated, as is the duplicate run. For example, in experiment 3, X_1 and X_2 fall within the constraint, whereas X_3, having a value of two, falls below the constraint of $5 \le X_3 \le 10$ and hence is eliminated. The results show three acceptable blends, plus the centroid, for four blends that should be included (at the very least) in the mixture experiment. The centroid is calculated by averaging X_1, X_2, and X_3 in each of the three acceptable experiments.

DESIGN OF EXPERIMENT

Using design-of-experiments (DOE) software, the author identified the extreme vertices (blends) in order to push the limits of the existing formulation. A constrained mixture design experiment was chosen, keeping fiber, metal powder, and filler weight percentages constant while varying binder, friction modifier, and lubricant. Table 2 shows the nine constants.

TABLE 2: Nine of the twelve factors were held constant in the new formulation, making up 70 weight percent of the brake lining.

MATERIAL	WEIGHT PERCENT
Fiber 1	10
Fiber 2	20
Metal powder	7
Six Various Fillers	33
TOTAL	**70**

The remaining three components were allowed to vary within the remaining 30 weight percent needed to bring the total to 100 percent. Components X_1 (binder), X_2 (friction modifier), and X_3 (lubricant) and their constraints are shown in Table 3.

TABLE 3: Factors and their constraints chosen for experimentation in the new formulation. These three components must add up to 30 weight percent. When combined with the nine factors totaling 70 weight percent held constant in Table 2, the full formulation equals 100 weight percent.

FACTOR	CONSTRAINT IN WEIGHT PERCENT
Binder (X_1)	$13 \le X_1 \le 15$
Friction modifier (X_2)	$10 \le X_2 \le 15$
Lubricant (X_3)	$5 \le X_3 \le 10$
TOTAL	**30**

Due to the geometric nature of experimental regions, ambiguities often exist when trying to identify extreme blends. The first step in removing these uncertainties is to enumerate all combinations stemming from the constraints, as shown in Table 3. In mixture design, when there are q number of components, the number of combinations is $q*2^{q-1}$, so here the total number of potential blends is $3*2^2$, or twelve. However, as you will see, some of these

The resulting four experimental mixture design blends, now shown as MD_1 through MD_4, make up Table 6.

TABLE 5: Experiment set up after substituting lower and upper level values of factors X_1, X_2, and X_3. Violated constraints, duplicates, and their associated experiments are struck through and shown in red.

EXPERIMENT	BINDER (X_1)	FRICTION MODIFIER (X_2)	LUBRICANT (X_3)
1	13	10	7
2	15	10	5
~~3~~	13	15	~~2~~
~~4~~	15	15	~~0~~
5	13	12	5
~~6~~	15	~~5~~	10
~~7~~	13	~~7~~	10
~~8~~	15	~~5~~	10
~~9~~ (Duplicate)	~~15~~	~~10~~	~~5~~
~~10~~	~~10~~	15	5
~~11~~	~~10~~	10	10
~~12~~	~~5~~	15	10
Centroid	13.67	10.67	5.67

TABLE 6: Experiment setup after eliminating repetitions and constraint violations.

EXPERIMENT	X_1	X_2	X_3
MD_1	13	10	7
MD_2	15	10	5
MD_3	13	12	5
MD_4	13.67	10.67	5.67

Table 7 lists the material compositions of all components, making up 100 weight percent of the brake lining material. Table 8 compares quality characteristics of the original base formulation and the objectives for the new formulation.

RESULTS AND DISCUSSION

Calculations were carried out using Design-Expert® software from Stat-Ease, Inc. Because there were only four unique blends, a linear model was chosen. (For three components, at least six unique blends are required to fit a curvilinear model. However, the centroid blend (MD_4) provides an indication of potential curvature. For example, it may exhibit a significantly higher response than expected, thus indicated in the presence of a peak. If so, a follow up experiment would be warranted to fine-tune the mixture.)

Based on this predictive model, the DOE software suggested a number of combinations, shown in Table 9, that would meet or exceed the required quality characteristics. The composition of all other nine materials remains constant at 70 weight percent.

TABLE 7: Material composition weight percents after reincorporating the constants.

MATERIAL	BASE	MD_1	MD_2	MD_3	MD_4
Fiber 1	5	10	10	10	10
Fiber 2	20	20	20	20	20
Metal powder	9	7	7	7	7
Binder (X_1)	12	13	15	13	13.67
Friction modifier (X_2)	5	10	10	12	10.67
Lubricant (X_3)	5	7	5	5	5.67
Six Various Fillers	44	33	33	33	33
TOTAL	**100**	**100**	**100**	**100**	**100**

TABLE 8: Comparison of base formulation and experiment objective quality characteristics.

QUALITY CHARACTERISTIC	UNIT	BASE	OBJECTIVE
Specific gravity	---	2.15	1.85 - 1.95
Rockwell hardness	R Scale	112	70 -100
Cold compressibility	%	0.39	0.75 min
Coefficient of friction (Mue)	--	0.50	0.35 - 0.45
Wear	mm	0.52	0.40 max

TABLE 9: Predicted values of quality characteristics using point prediction from the DOE software.

MIXTURE	X_1	X_2	X_3	SPECIFIC GRAVITY
1	13.00	10.67	6.33	1.921
2	13.25	10.67	6.08	1.923
3	13.50	10.67	5.83	1.925
4	13.75	10.67	5.58	1.927
5	14.00	10.67	5.33	1.929
6	14.25	10.67	5.08	1.931
7	14.50	10.50	5.00	1.936
8	14.75	10.25	5.00	1.942
9	15.00	10.00	5.00	1.949

Table 10 shows physical and frictional property comparisons between the base formulation and a confirmatory mixture experiment where $X_1=13.50$ weight percent, $X_2=10.67$, and $X_3=5.83$. You can see that quality characteristics improved dramatically over the base formulation and fell within the objectives.

TABLE 9: Predicted values of quality characteristics using point prediction from the DOE software (continued).

ROCKWELL HARDNESS	COMPRESSIBILITY	COEFFICIENT OF FRICTION (Mue)	WEAR
89.60	0.873	0.398	0.383
90.35	0.857	0.395	0.377
91.10	0.841	0.392	0.370
91.85	0.825	0.389	0.364
92.60	0.808	0.386	0.358
93.35	0.792	0.383	0.352
95.02	0.756	0.375	0.339
97.14	0.710	0.364	0.323
99.27	0.663	0.353	0.306

TABLE 10: Comparison of predicted quality characteristics of confirmatory experiment with the base formulation and mixture design objective.

QUALITY CHARACTERISTICS	UNIT	BASE	OBJECTIVE	CONFIRMATORY
Specific gravity	---	2.15	1.85 - 1.95	1.925
Rockwell hardness	R Scale	112	70 -100	91.10
Cold compressibility	%	0.39	0.75 min	0.841
Coefficient of friction (Mue)	--	0.50	0.35 - 0.45	0.392
Wear	mm	0.52	0.40 max	0.370

3D response surface plots reveal how quality characteristics are distributed within a design space. Using analysis of variance (ANOVA), the final equation of the response for actual component wear is:

$$Wear = (-0.015625 * X_1) + (0.049375 * X_2) + (9.37500\text{E-}003 * X_3)$$

Substituting X_1, X_2, and X_3, we can obtain responses for any given point in the design space. Plotting the corresponding values, the 3D representation of wear in the design space is shown in Figure 1. In the same way, we can look at 3D plots for any response by substituting the vales of X_1, X_2, and X_3 in the corresponding response equations. For instance, Figure 2 shows the 3D representation of Rockwell hardness.

The mixture contours for wear and Rockwell hardness are shown in Figure 3 and Figure 4, respectively. From the prediction, we can determine mixture components as well as confidence levels above and below the predicted values.

Substituting the values of $X_1=13.42$, $X_2=11.17$, and $X_3=5.41$ in the wear equation, we get the response: wear = 0.39, as shown in the box in Figure 3. The contour for predicted wear is also shown. Using corresponding equations, we can view plots for other responses.

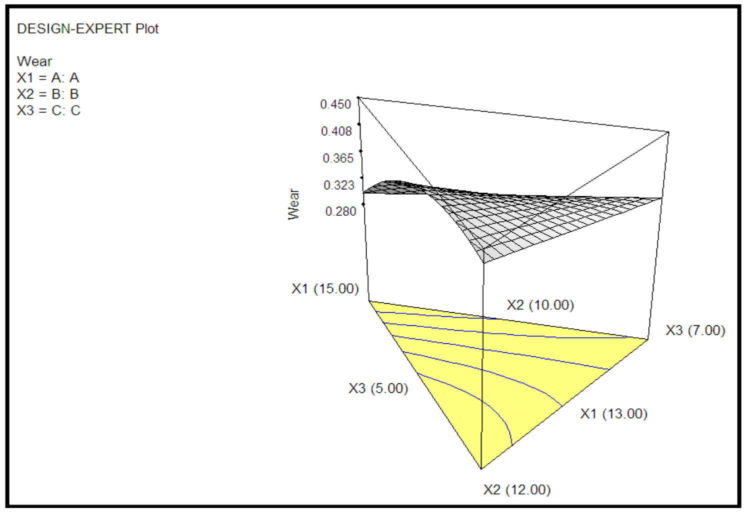

FIGURE 1: 3D response surface for wear *(Graph courtesy of Stat-Ease, Inc.)*

Similar to the wear calculation, Rockwell hardness (RH) can be calculated as,

$$RH = (6.64222 * X_1) - (1.85778 * X2) + (3.64222 * X_3)$$

Substituting the values of $X_1=13.39$, $X_2=10.75$, and $X_3=5.66$ in the equation for Rockwell hardness, we obtain 90.89 in the box in Figure 4.

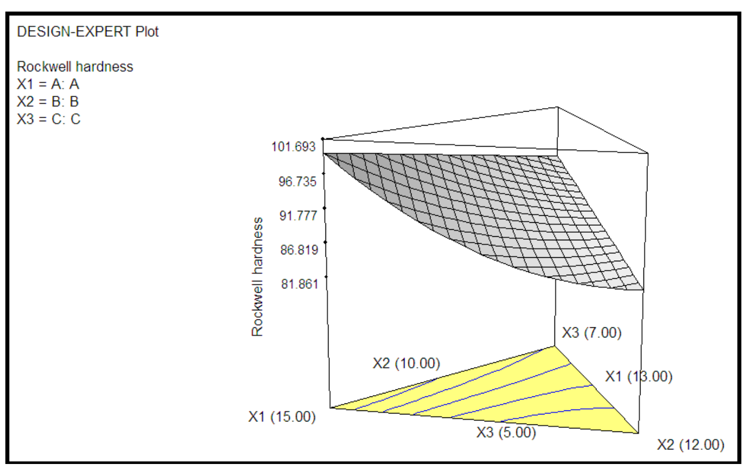

FIGURE 2: 3D response surface for Rockwell hardness. *(Graph courtesy of Stat-Ease, Inc.)*

An XY plot for quality characteristics is shown in Figure 5. From this plot, we can find the optimized values for the quality characteristics as

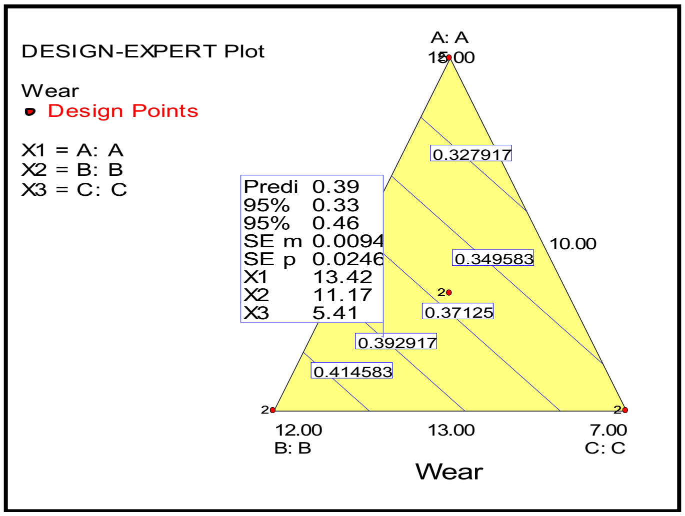

FIGURE 3: Mixture contour plot for wear. *(Graph courtesy of Stat-Ease, Inc.)*

well as the mixture components. To easily view the allowed minimum and maximum limits, the plot was generated using graphical optimization capabilities within Design-Expert DOE software. The resulting software-generated graph highlights the area of operability. By choosing any point in the design window, one can obtain in-specification response values corresponding to any X_1, X_2, and X_3 mixture. For example, in Figure 5, the values of specific gravity (1.9135), compressibility (0.9226), coefficient of friction (mue) (0.4119), and wear (0.3982) are given in the box corresponding to X_1=13.34, X_2=11.26, and X_3=5.40.

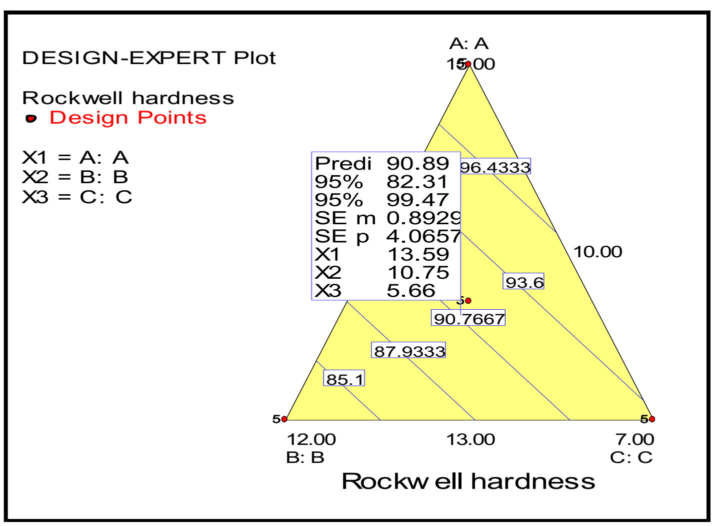

FIGURE 4: Mixture contour plot for Rockwell hardness. *(Graph courtesy of Stat-Ease, Inc.)*

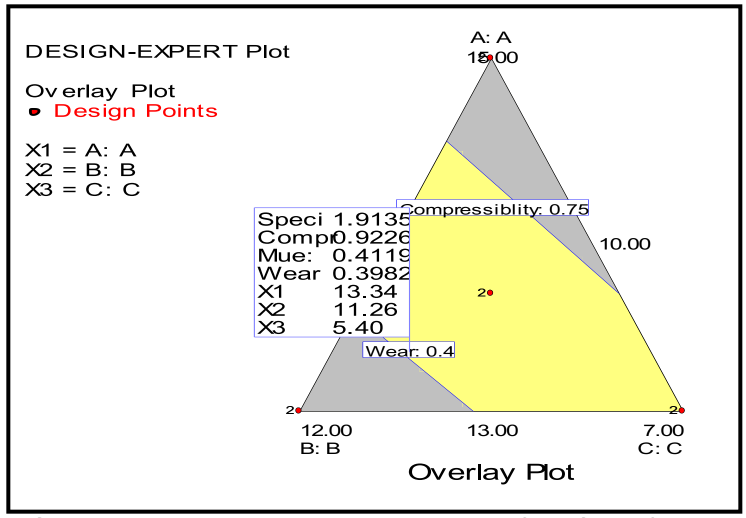

FIGURE 5: XY plot and prediction for Specific Gravity, Compressibility, Coefficient of friction (Mue), and Wear. *(Graph courtesy of Stat-Ease, Inc.)*

CONCLUSION

Binder concentration was critical when determining physical, frictional, and wear characteristics for this brake lining. Based on point predictions made possible by an extreme vertices mixture design aided by DOE software, binder concentration was selected at 13.50 weight percent, friction modifier at 10.67 weight percent, and lubricant at 5.83 weight percent. By following the tutorial nature of this case study, readers learn how to perform reformulations for their own needs.

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