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Introduction

Companies and service organizations worldwide have become aware of the New Economic Era (1), focused on quality and productivity improvement as the best strategy to survive in today's marketplace. The idea that quality is achieved by constantly improving all processes and products on a companywide basis (2–4) is gaining ground in many organizations.

Statistics is a key ingredient of Quality Leadership Management (5), for if decisions are to be based on data instead of guesswork, the use of a scientific approach becomes standard procedure. In many instances, only elementary statistical tools (6) will be required to achieve substantial improvements, especially if these tools are applied with constancy of purpose and with genuine top management involvement. On the other hand, in the domain of new product and/or process design, development, and improvement, the more complicated statistical techniques of design of experiments (7–10) and quality engineering (11) are very useful, especially if the good and new ideas contained in Ref. 11 are applied with good statistical methods as has been suggested in Ref. 12.

As has been pointed out (13), experimentation in the complex world of industrial and service organizations is much more than deciding on a matrix of experimental points. In our opinion, a deep understanding of the basic engineering concepts underlying the process being studied as well as its technical and economic constraints, together with a profound knowledge of statistics, are necessary conditions for successful real world experiments.

The experimental design described here is a plant experiment and was run in order to improve the process of manufacturing a particular brand of pet food. Several responses were measured, for the goal was not only to improve quality but quantity and cost as well. The process engineer, the quality assurance engineer, two process operators, together with the authors of this report, were the members of the quality improvement team.

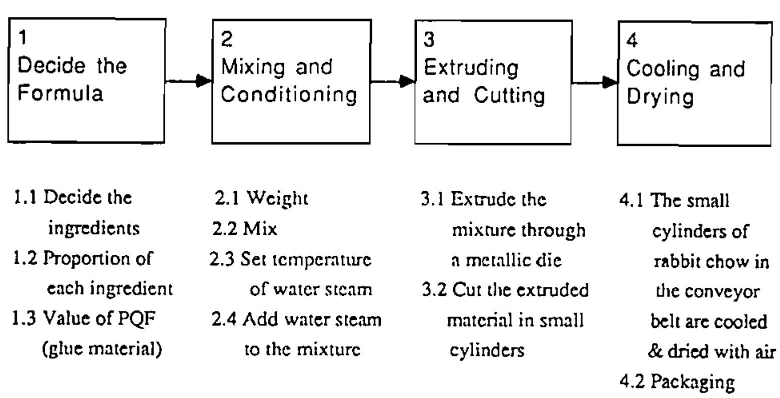
This article defines the problem, tells why it was important, describes the process of developing a solution, and provides a partial explanation of the results.

The Problem

The process of manufacturing rabbit food is schematically represented in the topdown flowchart (5) of Figure 1.

The two main quality-related problems in the process were:

1. During the cooling and drying of the rabbit food pellets, a loss of product in the form of fine powder was taking place. Reducing this kind of loss was one objective.
2. The most important problem from the point of view of the customer was that after packaging, during manipulation and transportation, the pellets eroded,



leaving a fine powder residue. This created digestive problems in the rabbits in addition to representing a loss of useful product.

The first problem (amount of powder in the process) could be easily measured as percentage of the process yield. The second problem (amount of powder at the customer's point of purchase) was not measurable at the manufacturing plant directly. Therefore, the process of the product's erosion during transportation was simulated in two special pieces of equipment called "checkers." During every run (batch), 4 samples of 3 kg each were taken at 5 minute intervals. Each sample was then divided into two parts which were submitted to the checkers for a period of 30 minutes. The response, the average of eight observations, was measured as percentage of powder in 1.5 kg of product.

Although the main objective was to improve quality, the engineers were also interested in controlling productivity and cost. Therefore, two additional factors were considered. Productivity was measured by the process yield in Tm/hour (metric tons/hr) and cost was considered in the form of energy consumption in each batch.

Broadly speaking, our objective was to improve quality, especially with the aim of minimizing the amount of powder or dust in the product while keeping productivity, losses of product during the process, and cost at acceptable levels. In addition to this immediate objective, the quality improvement team had other more intangible purposes in mind. Among them:

1. To educate the company to consider quality from the customer's point of view (market-in) instead of quality from an internal perspective (product-out). The fact that for the first time the amount of dust in the product arriving at the point of consumption was considered is a step in the right direction.
2. To show the engineers and plant personnel how simple experiments permit correlation of the final quality, productivity, and cost characteristics to controllable variables in the process, thus allowing the standardization of the operating conditions.
3. To educate plant personnel in the necessity of collecting good data for decision making. In this sense, the fact that only 12 sets of data obtained in a single day supplied a great amount of information on the process was an excellent way of conveying the message.
4. To educate the engineers in the usefulness of scientific feedback arising from comparing data with theories or hunches.

Factors and Constraints

Although as we will see below, the experiment was run in a single day, it took several weeks of work on the part of the quality improvement team to prepare the design and to instruct all those involved. First, the plant manager had to be convinced of the importance of running in-plant experimental designs in order to improve products and processes. This was accomplished through a two-hour presentation following a two-day detailed visit to the plant in order to obtain on-site information of what the problem was, the physical layout of the process, the constraints involved, and other relevant knowledge of the plant's operation. Second, we pointed out to the engineers that if one wants to get useful information with a very limited number of runs, it is essential to guarantee the quality of the data to be gathered during the experiment. They proceeded to train the operators and to make capability studies of the checkers and other measurement instruments. These studies convinced us that the measurement error was negligible compared with the noise in the process.

In order to facilitate data collection, we prepared a special form for the plant operators, containing detailed information on the run order, the values at which every experimental factor should be set, space for recording all the responses in each run, and as Box (17) recommends, in order to allow for Murphy's law, space was allocated for recording any incidents that occurred during the experimentation period.

In order to determine which factors affected the quality, productivity, and cost characteristics, we asked the engineers the following question: What do you do, during daily operation of the process, when quality deteriorates? (Note: powder is the only observable measure of quality).

They answered:

First, reduce the flow of mixture in the extrusion (step 3), although this will reduce yield.

Second, raise the conditioning temperature of the mixture in (step 2.3). This will increase the energy consumption.

Third, change the compression length of the die used in step 3 (extrusion). This is very time consuming and reduces yield.

Finally, as a last resort, one may change the formula by adding glue material (step 1.3).

During the meeting at the manufacturing plant, following a detailed first-hand experience with the process, it was suggested that mixture-type designs could be applied in order to study the quality impact of different formulas. This idea, which we may use in the future, was discarded in the first experimental design because it would increase the necessary number of runs to an unacceptable level.

Therefore, for reasons of economy and from our engineering knowledge of the process, it was decided to experiment with four factors, each at two levels. The factors and levels selected were those of Table 1.

The levels of the factors were chosen according to the following criteria: the experiments had to be run in the real plant and salable product ought to be produced. Also, all runs should be performed during one day. This implied a maximum allowable number of 13 runs.

Table 1. Factors and Levels

	FACTOR	LEVEL −	LEVEL +
X_1:	Formula (PQF)	10	20
X_2:	Conditioning temperature	80% of T (°C)	T (maximum)
X_3:	Flow	80% of F (Tm/h)	F (maximum)
X_4:	Compression zone in die	2″	2½″

It was also decided that no confounding between main effects and two-factor interactions was admissible. The reason was that, in a multiple response situation, different unexpected interactions could affect each response and, therefore, at least all two-factor interactions should be considered. This excluded the possibility of running a 2^{4-1} fractional factorial or a 2^3 complete factorial; even if one could assume that only a few factors are important for each response, those factors could differ from one response to another and therefore all four factors should be considered.

Finally, randomization was discussed. It was suggested that factor 4 (compression zone of the die) was difficult to change. We presented the engineers with two possibilities. First, one could consider a split-plot type of design with factor 4 confounded with the main plots. The problem with this design is that the effect of any "lurking variable" (10) that could affect the responses is going to be confounded with factor 4. Another alternative was to completely randomize the design. As a trade-off, it was decided to change factor 4 three times during the day.

Experimental Design

All of the above-mentioned constraints could be accommodated in a 12-run nonorthogonal design of resolution V, of the type described in Ref. 14. The design is presented in Table 2, where the responses are:

Y_1 : Powder in the product
Y_2 : Powder in the process
Y_3 : Yield
Y_4 : Energy consumption

In Appendix 1, we show that this design is of resolution V. This means that we can estimate all four main effects and all the two-factor interactions provided that interactions of order three and four are unimportant (as is usually the case).

If one were not sure that higher order interactions are negligible, then a full factorial design with 16 runs should be considered.

Table 2. The Experimental Design (a 3/4 Fraction of a 2^4 and the Responses)

RANDOM ORDER	RUN NUMBER	FACTORS				RESPONSES			
		X_1	X_2	X_3	X_4	Y_1	Y_2	Y_3	Y_4
12	1	−	+	−	+	.916	1.92	7.50	222.5
9	2	+	−	−	−	1.178	2.07	8.70	238.0
3	3	−	+	+	−	1.216	1.85	10.20	250.4
4	4	+	−	+	+	1.119	2.03	6.20	250.4
1	5	−	−	−	−	1.315	1.66	8.30	235.0
10	6	+	+	−	+	.911	2.08	7.20	222.0
11	7	−	−	+	+	1.070	1.96	7.95	267.5
2	8	+	+	+	−	1.273	2.13	9.60	248.2
8	9	−	+	−	−	1.071	1.62	8.50	224.0
6	10	+	−	−	+	1.025	1.73	5.90	233.3
5	11	−	+	+	+	1.040	1.64	7.30	248.5
7	12	+	−	+	−	1.174	1.93	9.95	255.0

Analysis

Although scatterplots between pairs of responses indicated the existence of correlation between some responses, we proceeded to the analysis of each response separately. The correlation was taken into account, at the end of the study, by looking at all responses jointly.

For each Y_i ($i = 1,2,3,4$), we fitted a model including the four main effects and the six two-factor interactions by least squares. Then, the estimated effects were plotted on normal probability plots and a simplified model, including only the significant effects, was again fitted by least squares.

Several graphical diagnostic tools were used in order to check the models, and in searching for possible outliers and/or influential observations.

The analysis for the response of main interest, Y_1 (powder in the product) as well as the main results for Y_2, Y_3, and Y_4 are presented in this section.

The estimated coefficients in the model:

$$Y_{i1} = \beta_0+\beta_1X_1+\beta_2X_2+\beta_3X_3+\beta_4X_4+\beta_{12}X_1X_2+\beta_{13}X_1X_3+ \beta_{14}X_1X_4+\beta_{23}X_2X_3+\beta_{24}X_2X_4+\beta_{34}X_3X_4+\epsilon_i \quad (1)$$

are:

μ	X_1	X_2	X_3	X_4	X_1X_2	X_1X_3	X_1X_4	S_2X_3	X_2X_4	X_3X_4
b_0	b_1	b_2	b_3	b_4	b_{12}	b_{13}	b_{14}	b_{23}	b_{24}	b_{34}
1.12	−.0045	−.03613	.0449	−.0674	.0249	.0310	.0155	.0534	.0001	.0096

These effects are plotted in Figure 2, after excluding b_0.

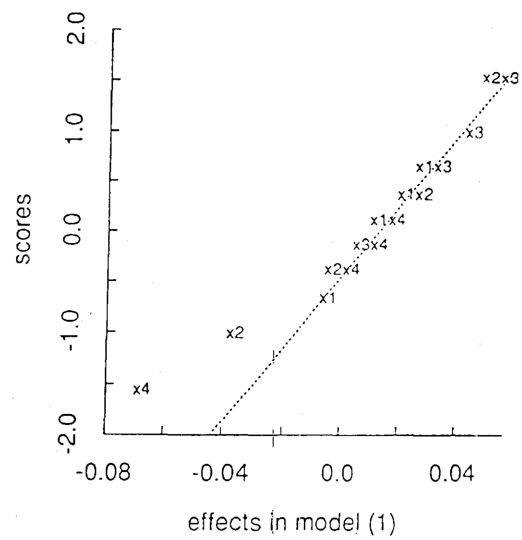


Figure 2. Normal probability plot of estimated effects in model (1). Response is powder in the product.

Only X_4 has clearly a significant effect on the mean and X_2 might also be of some importance, therefore the tentative model:

$$Y_{1i} = \beta_0 + \beta_2 X_2 + \beta_4 X_4 + \epsilon_{1i} \tag{2}$$

was fitted by ordinary least-squares analysis. The estimated coefficients and standard errors are:

Factor :	μ	X_2	X_4
Coefficient :	b_0	b_2	b_4
Value :	1.109	$-.038$	$-.095$
Standard error :		.022	.022

The plot of residuals versus the predicted values, $\hat{Y}$, showed no unusual pattern, and model (2) was tentatively retained.

Projecting the data for response Y_1, in the X_2X_4 plane one gets Figure 3.

The effect of X_4 is quite clear. In fact, the model containing only this factor is almost as good as model (2) (see Fig. 4).

This model is:

$$\hat{Y}_1 = 1.109 - .0955\, X_4 \tag{3}$$

Standard error = (.0244)

The conclusion is then that only the compression zone of the die has an important effect on the main quality characteristic, with a small additional effect of the conditioning temperature. This is consistent with β_2 being barely significant in model (2).

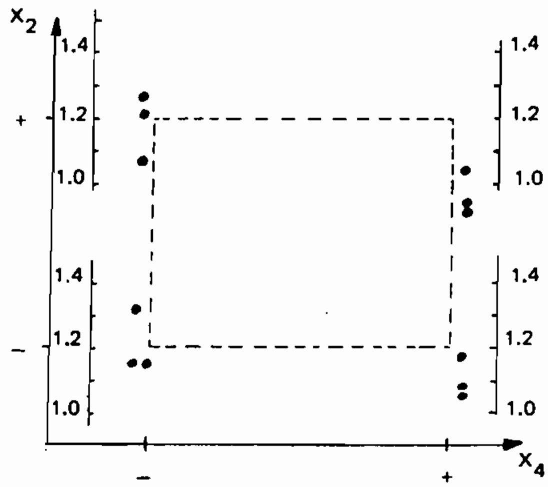


Figure 3. Response Y_1 projected in the X_2, X_4 plane.

Following the same approach, the best model for Y_2, powder in the process, was found to be:

$$\hat{Y}_2 = 1.885 + \underset{(.043)}{.11X_1} \tag{4}$$

Therefore, this response is only affected by the PQF (glue material) contained in the formula.

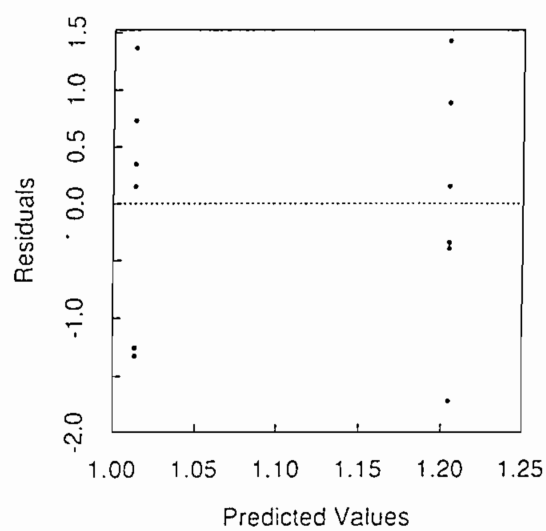


Figure 4. Standardized residuals, e_i, versus predicted values, Y_i, in Model (3).

When modelling the response Y_3 (process yield), an outlier was detected. The normal probability plot of the estimated effects for the model:

$$Y_3 = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \cdots + \beta_{34} X_3 X_4 + \epsilon_3 \quad (6)$$

appears on Figure 5.

As has been pointed out (16), outliers can be identified in half-normal probability paper. In a normal probability plot the split around 0 could be an indication of an outlying observation. One way to detect this possible outlier is to compute

$$\mathbf{H} = (\mathbf{X}'_1 \mathbf{X}_1)^{-1} \mathbf{X}_1$$

for $\mathbf{b} = \mathbf{H}\mathbf{y}$. The signs of the columns of $\mathbf{H}$ are the signs that each individual observation has in the linear contrasts for each effect. In our case, the observation corresponding to run #10 had, minus sign in the contrasts for X_3, X_1X_2, X_2, and X_2X_4 and therefore could be responsible for the observed split. In fact, the value of this observation, 5.90, was found to have been incorrectly copied in the data sheet, the true value being 6.90.

In the corrected plot of Figure 6, only X_4 seems to affect yield. The model was:

$$\hat{Y}_3 = 8.19 - \underset{(.2045)}{1.0179} X_4 \quad (7)$$

and no problems were detected in the residuals.

Finally, when modelling the energy consumption, observation #7 was also a suspected outlier. No reason was found for this observation, so that it was retained in a first analysis.

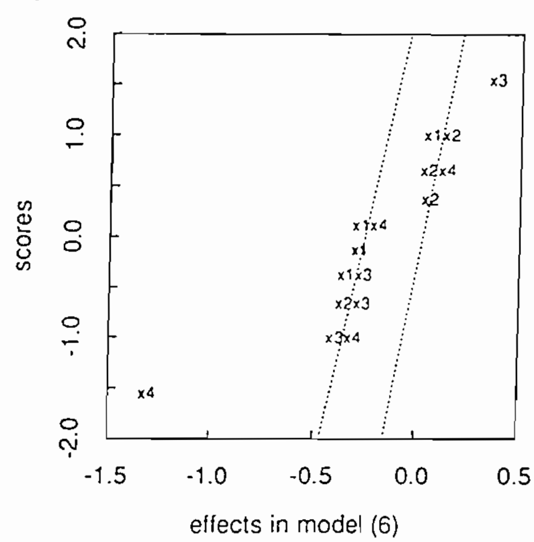


Figure 5. Normal probability plot of estimated effects in Model (6). Response in process yield.

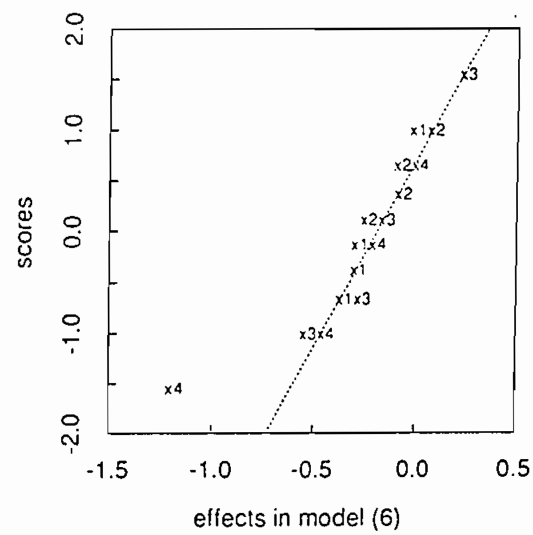


Figure 6. Normal probability plot for model (6) with observation #10 reset to the correct value.

First, we fitted the model:

$$Y_4 = \beta_0 + \beta_2 X_2 + \beta_3 X_3 + \beta_{23} X_2 X_3 + \epsilon_4 \tag{8}$$

The residual plot of this model appears in Figure 7. Again, observation #7 was pointed out as an outlier.

This observation was also a outlier in all the scatterplots of Y_4 versus the other responses. Because of accumulating evidence, run #7 was excluded in the estimation of the model (8). The results are:

$$Y_4 = 240 - 4.07\, X_2 + 10.87\, X_3 + 2.23\, X_2 X_3 \tag{9}$$
$$(.60)(.60)(.60)$$

and no problems appeared in the residual plot.

Main Results

The results of the analysis are summarized in Table 3.

The existence of interaction between factors X_2 and X_3 on energy consumption suggests that those factors should be analyzed jointly as in Figure 8.

In order to get scientific feedback (18), the models summarized in Table 3, and especially the *signs* with which the different factors appear, where analyzed from an engineering point of view. As in many real situations only partial explanations were possible.

For example, the fact that increasing the compression zone of the die (X_4) has a negative effect on the yield (Y_3) seemed reasonable due to an increase in the

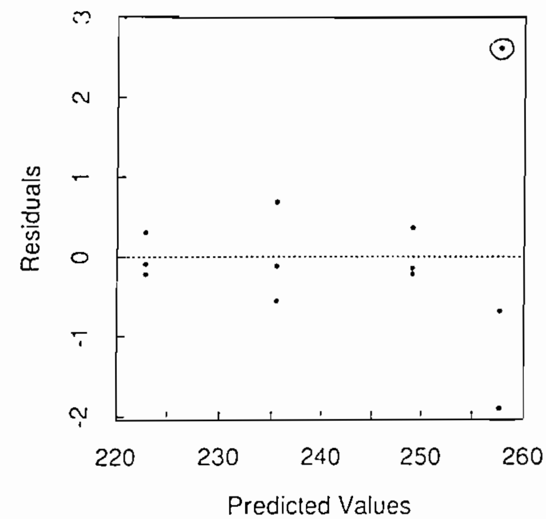


Figure 7. Standardized residuals versus fitted values in Model (8).

difficulty of extruding the mixture. Also, the increase in energy consumption (Y_4) when the flow (X_3) is increased was expected, but the fact that increasing the conditioning temperature (X_2) while keeping the flow rate (X_3) at its low level gives the lowest energy consumption deserves further engineering studies in order to explain this unexpected interaction. In fact, the engineers expected an increase in energy consumption when increasing the conditioning temperature as explained earlier.

Another surprise was the fact that the powder or dust in the process (Y_2) is not very well explained by the factors considered during the experiment. Only the glue material (X_1) has a barely significant effect and in the opposite direction as expected by the engineers. This also requires further thinking on the physics of the process.

Table 3. Summary of Results

	COEFFICIENTS FOR FACTORS:						
RESPONSE	CONSTANT	X_1	X_2	X_3	X_4	X_2X_3	ADJUSTED R^2
Powder Y_1 = in product	1.109		−.038		−.095		63.2%
Powder in Y_2 = in process	1.885	+.11					33.4%
Yield Y_3 =	8.19				−1.017		68.3%
Energy Y_4 = consumption	240		−4.07	10.87		2.23	97.5%

Observation #7 has been excluded for estimating the model corresponding to Y_4.

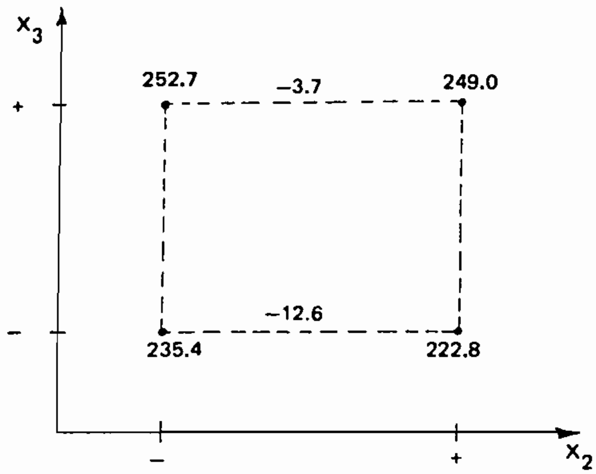


Figure 8. Mean energy consumption (Y_4).

Another lesson learned was that Y_2 (amount of powder in the process) is uncorrelated with all other responses. This can be seen in the bivariate scatterplots of Y_2 versus all other responses and also by noting that X_1 affects Y_2, but not Y_1, Y_3, or Y_4. Therefore, to use this indicator to control the quality characteristic Y_1 was not a practice to be recommended. In order to minimize the loss of useful product, Y_2 should be minimized. The recommendation was to use low levels of glue material X_1, or even better, try to run mixture type of experiments with the formula. This suggestion is going to be implemented in future experiments.

The main quality characteristic, Y_1 (amount of powder in the product) and the cost characteristic Y_4 (energy consumption), could be optimized simultaneously, but a trade-off was necessary between those responses and productivity, Y_3 (yield) involving the compression zone of the die. Using the high level of X_2 (conditioning temperature) and low level of X_3 (flow) decreases both the energy consumption and the amount of powder in the product, and therefore, those levels were recommended. Also, experimenting with lower levels of X_3 and higher levels of X_2 could be worth considering.

As for the compression zone, X_4, if used at its high level will increase quality (Y_1) but decrease productivity (Y_3). The reason for lower yield is that extrusion through a 2½″ die is more difficult than through a 2″ die.

The suggestion was made that, again, experimenting with the formula, one could try to reduce the viscosity of the extruded material and gain productivity. The question to be answered in future experiments is if quality, Y_1, can also be improved simultaneously.

Conclusions

Real plant experimentation faces the experimenter with economical and technical constraints. In this case study it has been possible to take into account those constraints in a 12-run resolution V design.

Four responses were studied. The internal losses in the process could be decreased in the short term by using low levels of glue material. Further mixture-type experiments were recommended with the formula in order to further decrease the internal losses as well as a way to solve the trade-off between quality and productivity arising from the effect of the compression zone of the die.

Finally, the conditioning temperature and the flow could be set in such a way that the quality improved while the energy consumption was also decreased.

Acknowledgments

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Appendix 1

One way to see that the design is of resolution V is as follows:

If the true model relating a response y_i to the experimental factors is:

$$\text{True model:} \qquad y_i = X_1\beta_1 + X_2\beta_2 + \epsilon_i$$

and we fit the model:

$$\text{Fitted model:} \qquad y_i = X_1\beta_1 + \epsilon_i$$

then if $b_1 = (\mathbf{X}'_1\mathbf{X}_1)^{-1}\mathbf{X}'_1\mathbf{Y}$ is the OLS estimation of $\beta 1$, it is well known that:

$$E(b_1) = \beta_1 + (\mathbf{X}'_1\mathbf{X}_1)^{-1}\mathbf{X}'_1X_2\beta_2 = \beta_1 + \mathbf{B}\beta_2$$

where **B** matrix is the bias matrix:

$$\mathbf{B} = (\mathbf{X}'_1\mathbf{X}_1)^{-1}\mathbf{X}'_1\mathbf{X}_2$$

If the four main effects and the six two-factor interaction are considered as being the columns of $\mathbf{X}_1$, and $\mathbf{X}_2$ has as columns the four three-factor interactions and the four-factor interaction then the product $\mathbf{B}\beta_2$ is:

$$
\begin{bmatrix}
0 & 0 & 0 & 0 & -1 \\
0 & 0 & 0 & 0 & -1 \\
-1 & 0 & 0 & 0 & 0 \\
0 & -1 & 0 & 0 & 0 \\
0 & 0 & -.33 & -.33 & 0 \\
0 & -1 & 0 & 0 & 0 \\
-1 & 0 & 0 & 0 & 0 \\
0 & -1 & 0 & 0 & 0 \\
-1 & 0 & 0 & 0 & 0 \\
0 & 0 & 0 & 0 & -1
\end{bmatrix}
\begin{bmatrix}
123 \\ 124 \\ 134 \\ 234 \\ 1234
\end{bmatrix}
$$

and therefore the confounding pattern, in the notation of Ref. 10, is:

$$
\begin{array}{ll}
1 = 1 - 1234 & 13 = 13 - 124 \\
2 = 2 - 1234 & 14 = 14 - 123 \\
3 = 3 - 123 & 23 = 23 - 124 \\
4 = 4 - 124 & 24 = 24 - 123 \\
12 = 12 - (.33)\,134 - (.33)\,234 & 34 = 34 - 1234
\end{array}
$$

which clearly shows the resolution V of the design. This high resolution in only 12 runs was achieved at the expense of orthogonality. The design is nonorthogonal as can be seen by computing the matrix $(\mathbf{X}'_1\mathbf{X}_1)^{-1}$. The relationship of the nonzero off-diagonal terms to the diagonal ones in this matrix is of the order: .06/.125 so that no severe nonorthogonality is present.

The matrix $(\mathbf{X}'_1\mathbf{X}_1)^{-1}$ is (where "$\cdot$" refers to 0)

$$
\begin{array}{c}
\begin{array}{cccccccccc}
1 & 2 & 3 & 4 & 12 & 13 & 14 & 23 & 24 & 34
\end{array} \\
\begin{bmatrix}
0.125 & 0.6 & \cdot & \cdot & \cdot & \cdot & \cdot & \cdot & \cdot & .06 \\
.06 & 0.125 & \cdot & \cdot & \cdot & \cdot & \cdot & \cdot & \cdot & .06 \\
\cdot & \cdot & 0.125 & \cdot & \cdot & \cdot & .06 & \cdot & .06 & \cdot \\
\cdot & \cdot & \cdot & 0.125 & \cdot & .06 & \cdot & .06 & \cdot & \cdot \\
\cdot & \cdot & \cdot & \cdot & 0.083 & \cdot & \cdot & \cdot & \cdot & \cdot \\
\cdot & \cdot & \cdot & .06 & \cdot & 0.125 & \cdot & .06 & \cdot & \cdot \\
\cdot & \cdot & .06 & \cdot & \cdot & \cdot & 0.125 & \cdot & .06 & \cdot \\
\cdot & \cdot & \cdot & .06 & \cdot & .06 & \cdot & 0.125 & \cdot & \cdot \\
\cdot & \cdot & .06 & \cdot & \cdot & \cdot & .06 & \cdot & 0.125 & \cdot \\
.06 & .06 & \cdot & \cdot & \cdot & \cdot & \cdot & \cdot & \cdot & 0.125
\end{bmatrix}
\end{array}
$$

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