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Report No. 37

**CASE STUDY: EXPERIMENTAL DESIGN  
IN A PET FOOD MANUFACTURING COMPANY**

Albert Prat\* and Xavier Tort\*\*

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\* Dr. Albert Prat is a Professor of Statistics at the School of Industrial Engineering, Polytechnic University of Catalonia in Spain. He has been a visiting Scholar at the Graduate School of Business, University of Chicago, and a visiting Professor at the University of Wisconsin-Madison. He is a member of I.S.I., A.S.Q.C., and A.S.A. This research was sponsored by the Vilas Trust of the University of Wisconsin-Madison and the Polytechnic University of Catalonia in Spain.

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**PRACTICAL SIGNIFICANCE**

Experimentation in the complex world of industry and service organizations requires a deep understanding of the basic engineering concepts underlying the process being studied, as well as relevant technical and economic constraints. The experimental design described in this paper is a plant experiment where those constraints were taken into account. Several responses were measured, for the goal was not only to improve quality but also to increase productivity and reduce cost.

Also the main steps of the problem solving strategy: what the problem was, why it was important, the process of developing a solution, as well as a partial description of the results, are presented in this case study.

**Key Words:** Quality Improvement, Design of Experiments, Fractional Factorials, Multiresponse, Non-orthogonal Designs

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# **CASE STUDY; EXPERIMENTAL DESIGN IN A PET FOOD MANUFACTURING COMPANY**

by

Albert Prat and Xavier Tort

## **1. Introduction**

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

Statistics is one of the key ingredients of the 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 in order 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 (11) are applied with good statistical methods as has been suggested in (12).

As has been pointed out in (13), experimentation in the complex world of Industry 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 in this paper 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 also quantity and cost. The process engineer, the quality assurance engineer, and two process

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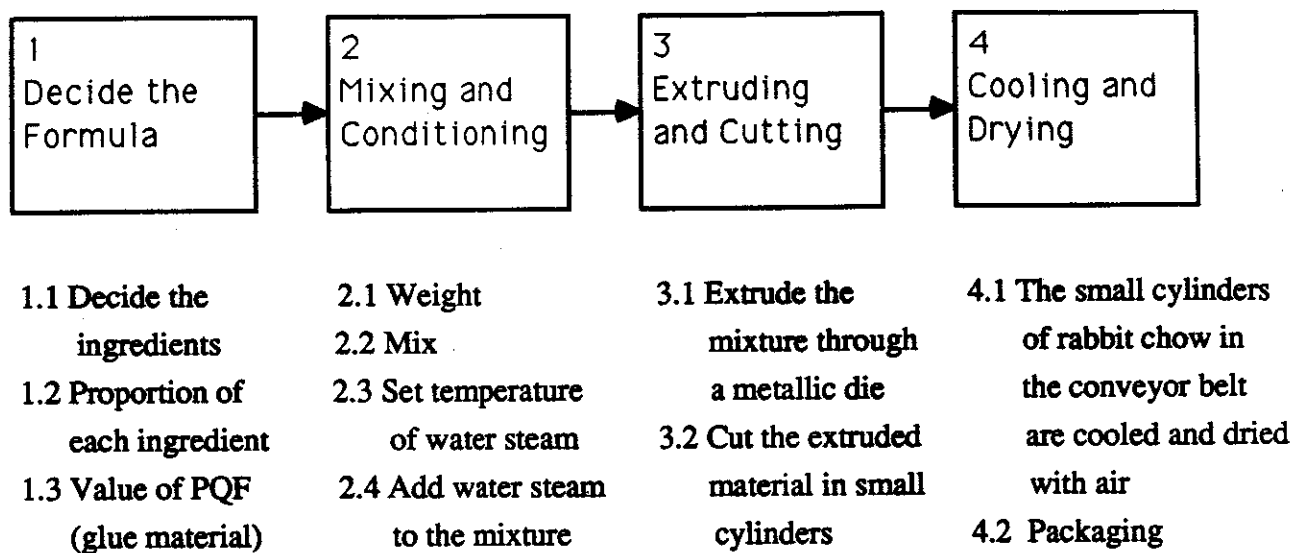
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operators, together, with the two authors of this paper, were the members of the quality improvement team.

This paper describes what the problem was, why it was important, the process of developing a solution, as well as a partial description of the results.

## 2. What the Problem Was:

The process of manufacturing rabbit's food is schematically represented in the top-down flowchart (5) of Figure 1.



**Figure 1.** Top-Down Flowchart of the Process.

The two main quality related problems in the process were:

- a) During the cooling and drying of the rabbit's food cylinders, a loss of product in the form of fine powder was taking place. Reducing this kind of loss was one of the objectives.
- b) The most important problem from the point of view of the customer was that after packaging, during manipulation and transportation, the cylinders eroded so that fine powder was produced. This created digestion problems in the rabbits in addition to representing a loss of useful product.

The first response (amount of powder in the process) could be easily measured as percentage of the process yield. The second response (amount of power arriving at the

customer's place) 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 kilograms (kg) each were taken at five minute intervals. Each sample was then divided into two parts that were submitted to the checkers for a period of 30 minutes. The response was the average of eight observations and it is 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 responses were considered. Productivity was measured by the process yield in Tm/hour (metric tones/hour) and cost was considered in the form of energy consumption in each batch.

Broadly speaking, our objective was to improve quality, especially 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:

- (i) Educate the company in considering quality from the customer's point of view (market-in) instead of quality from an internal prospective (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.
- (ii) To show to the engineers and plant personnel how simple experiments allow to relate the final quality, productivity and cost characteristics to controllable variables in the process, thus allowing the standardization of the operating conditions.
- (iii) Educate plant personnel in the necessity of collecting good data for decision making. In this sense, the fact that only 12 data obtained in a single day supplied a great amount of information on the process was an excellent way of conveying the message.
- (iv) Educate the engineers in the scientific feedback arising from company data with theories or hunches.

### **3. Factors and Constraints**

Although as we will see below, the experiment was run on a single day. It took several weeks of work of the quality improvement team to prepare the design and to instruct all people 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 after a two-day detailed visit to the plant in order to get on site information of what the problem was, the physical layout of the process, the constraints 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 as compared with the work in the process.

In order to facilitate data collection, we prepared a special form for the plant operators, with detailed information on the run order, the values at which every experimental factors should be set, space for recording all the responses in each run, and as George Box (17) recommends, in order to listen to Murphy, space was allowed for recording any incidence during the experimentation period.

In order to decide 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 that only powder in the process is observable).

In summary, their answer was:

– 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 resource, one may change the formula by adding glue material (step 1.3).

During the meeting held 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, although it is going to be pursued in the near future, was discarded in the first experimental design because it would increase the necessary number of runs to an unacceptable level.

Therefore, from economical considerations and from 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.

		LEVEL	
FACTOR		-	+
X <sub>1</sub> :	Formula (PQF)	10	20
X <sub>2</sub> :	Conditioning Temperature	80% of T	T (maximum)
X <sub>3</sub> :	Flow	80% of F	F (maximum)
X <sub>4</sub> :	Compression Zone in Die	2"	2 1/2"

Table 1. Factors and Levels.

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

It was also decided that no confounding between main effects and two factors 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 for 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" 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.

#### 4. The Experimental Design

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

Y<sub>1</sub>: Powder in the product

Y<sub>2</sub>: Powder in the process

Y<sub>3</sub>: Yield

Y<sub>4</sub>: Energy consumption

RANDOM ORDER	RUN NUMBER	FACTORS				RESPONSES			
		X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	X <sub>4</sub>	Y <sub>1</sub>	Y <sub>2</sub>	Y <sub>3</sub>	Y <sub>4</sub>
12	1	-	+	-	+	0.916	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	+	+	-	+	0.911	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

**Table 2.** The experimental design (a 3/4 fraction of a 2<sup>4</sup>) and the responses.

In Appendix 1, we show that this design is of resolution V. This implies 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 it is usually the case.

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

## 5. Analysis

Although scatter plots between pairs of responses as well as the given value analysis of  $D = (y - \bar{y})'(y - \bar{y})$  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 interaction by least squares. Then, the estimated effects were plotted in 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.

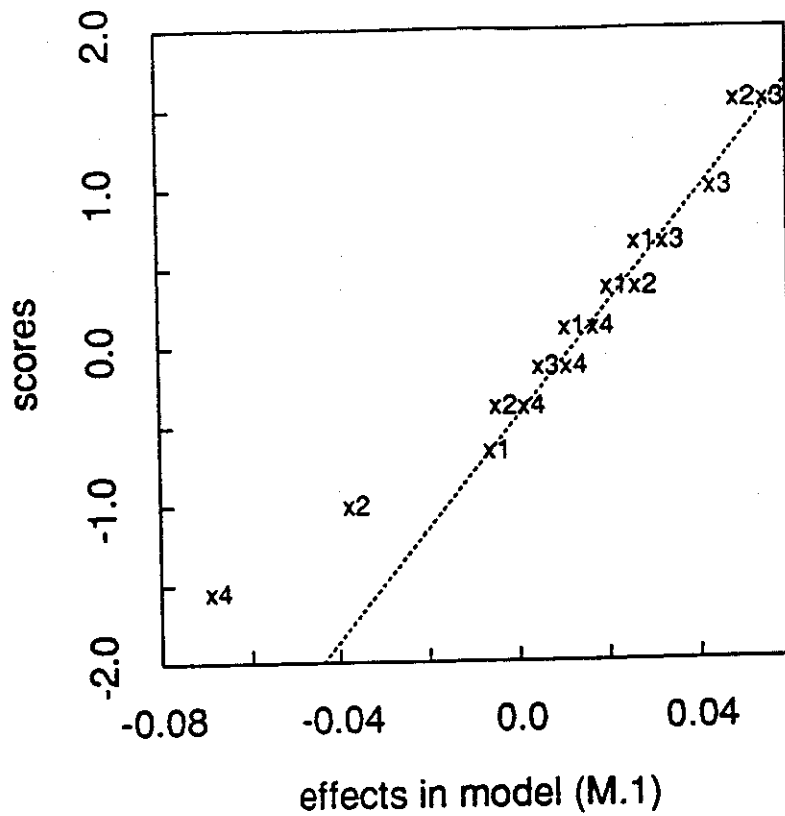
The estimated coefficients in the model

$$Y_{i1} = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_4 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{14} X_1 X_4 + \beta_{23} X_2 X_3 + \beta_{24} X_2 X_4 + \beta_{34} X_3 X_4 + \epsilon_i \quad (M.1)$$

are:

$\mu$	$X_1$	$X_2$	$X_3$	$X_4$	$X_1 X_2$	$X_1 X_3$	$X_1 X_4$	$X_2 X_3$	$X_2 X_4$	$X_3 X_4$
$b_0$	$b_1$	$b_2$	$b_3$	$b_4$	$b_{12}$	$b_{13}$	$b_{14}$	$b_{23}$	$b_{24}$	$b_{34}$
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 (M.1).  
Response is powder in the product.

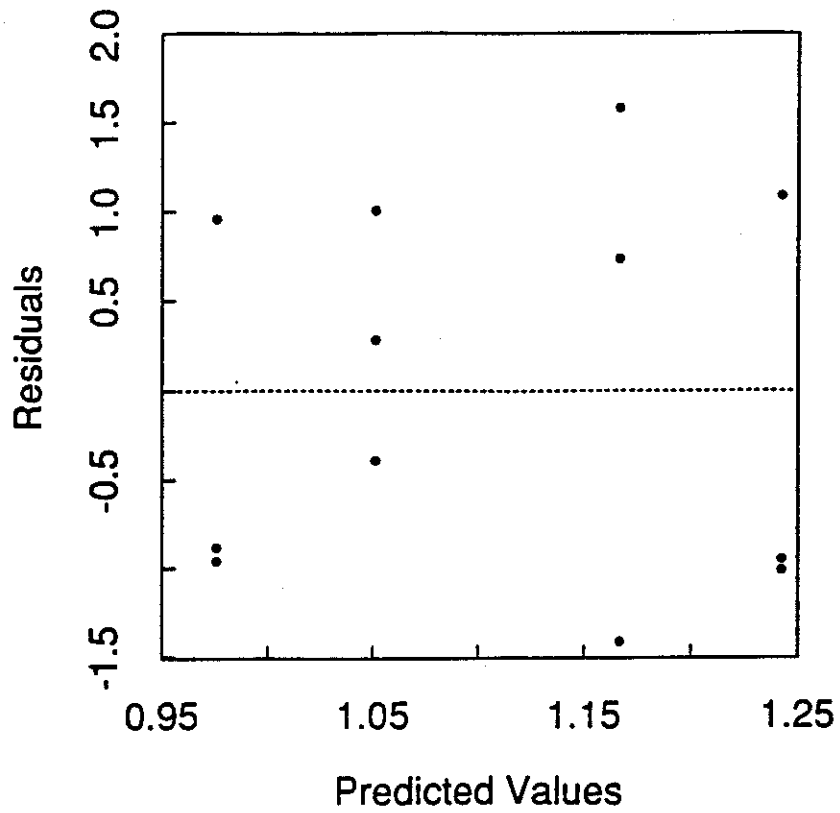
Only  $X_4$  and  $X_2$  seem to have a significant effect on the mean, therefore the model:

$$Y_{1i} = \beta_0 + \beta_2 X_2 + \beta_4 X_4 + \varepsilon_{1i} \quad (M.2)$$

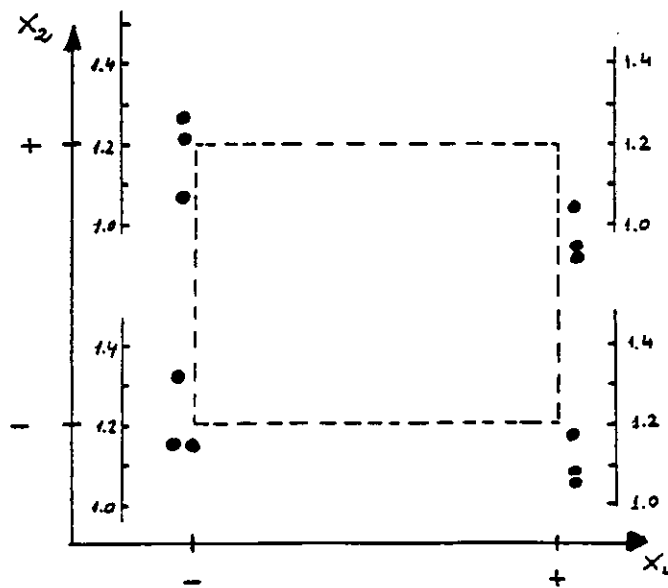
was fitted by ordinary least squares. The estimated coefficients and standard errors are:

Factor	$\mu$	$X_2$	$X_4$
Coefficient	$b_0$	$b_2$	$b_4$
Value	1.109	-.038	-.095
Standard Error		.022	.022

The standardized residuals,  $e_i$ , are plotted versus the predicted value,  $\hat{Y}$ , in Figure 3. No recognizable pattern was seen in this plot, and model (M.2) was tentatively retained.



**Figure 3.** Standardized residuals,  $e_i$ , versus predicted values,  $\hat{Y}$ , in model M.2. Projecting the data for response  $Y_1$ , in the  $X_2$   $X_4$  plane one gets Figure 4.



**Figure 4.** Response  $Y_1$  projected in the  $X_2$ ,  $X_4$  plane.

The effect of  $X_4$  is quite clear. In fact, the model containing only this factor is almost as good as M.2 (see Figure 5).

This model is:

$$Y_1 = 1.109 - .0955 X_4 \quad (M.3)$$

(.0244) = Standard Error

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  $\beta_2$  being barely significant by the t-test in model (M.2).

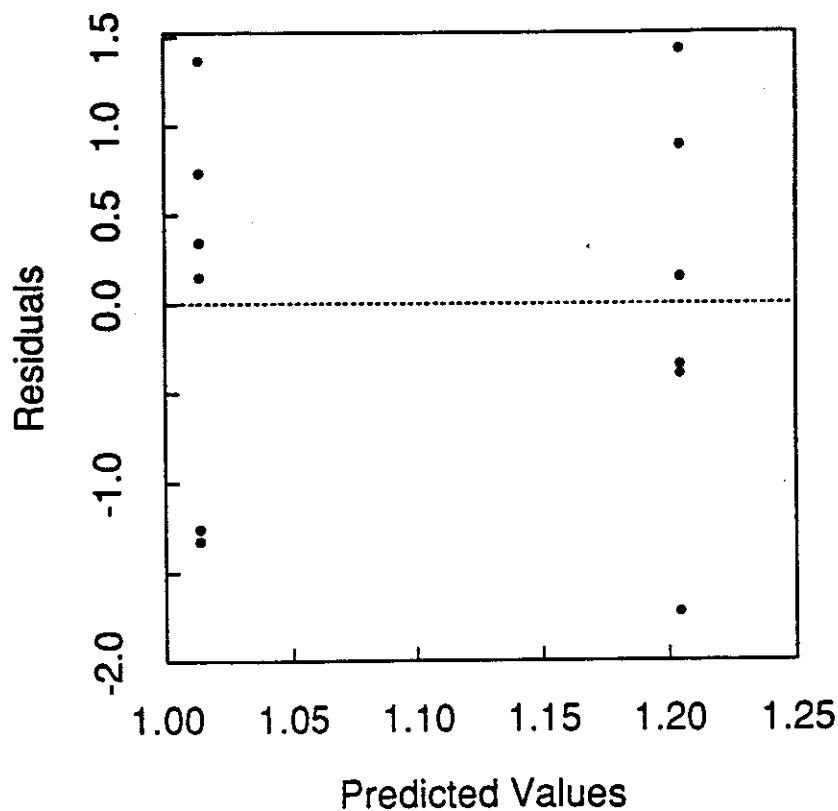


Figure 5. Standardized residuals,  $e_i$ , versus predicted values,  $\hat{Y}_i$ , in Model M.3.

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

$$\hat{Y}_2 = 1.885 + .11X_1 \quad (M.4)$$

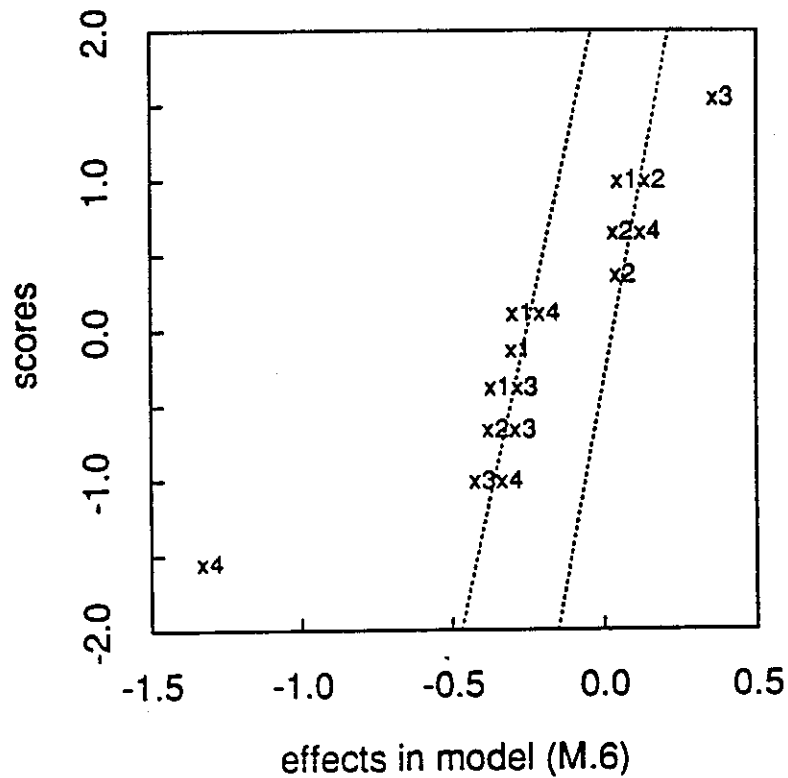
(.043)

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

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

$$Y_3 = \beta_0 + \beta_2 X_1 + \beta_2 X_2 + \dots + \beta_{34} X_3 X_4 + \varepsilon \quad (M.6)$$

appears on Figure 6.



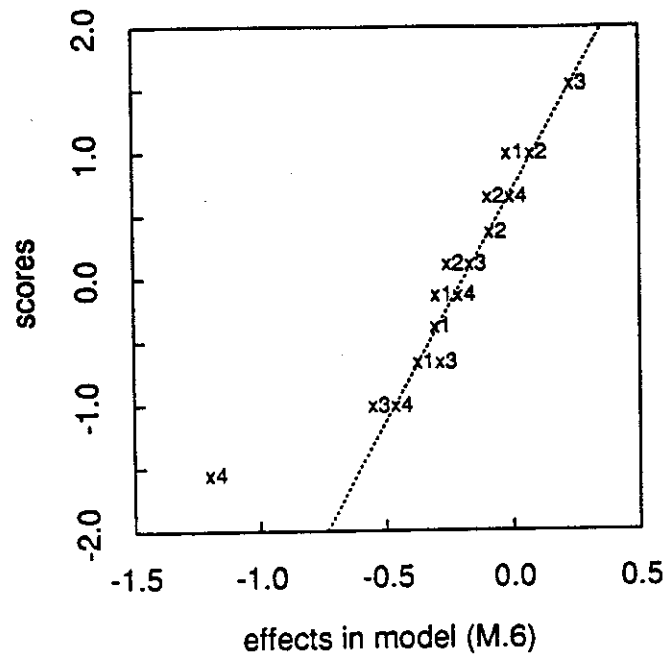
**Figure 6.**

Normal probability plot of estimated effects in model (M.6). Response is process yield.

As has been pointed out in (16), the split around 0 could be an indication of an outlying observation. One way to detect this possible outlier is to compute

$$H = (X_1'X_1)^{-1} X_1'$$

for  $b = Hy$ . The signs of the columns of  $H$  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.



**Figure 7.** Normal probability plot for model (M.6) with observation #10 reset to the correct value.

In the corrected plot of Figure 7, only  $X_4$  seems to affect yield. The model was:

$$\hat{Y}_3 = 8.19 - 1.017 X_4 \quad (M.7)$$

(.2045)

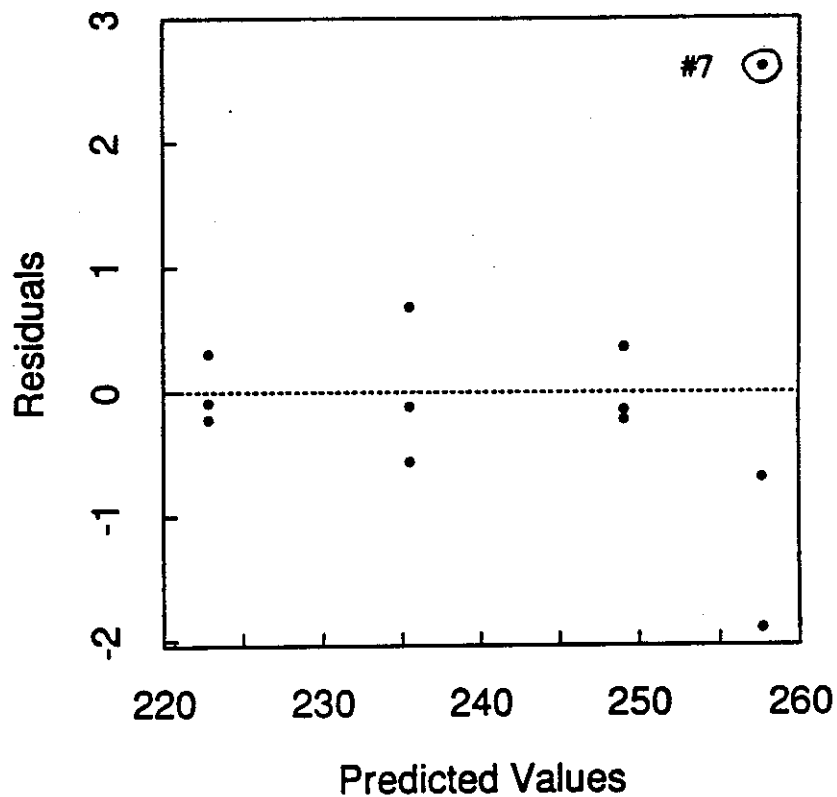
and no problems were detected in the residuals.

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.

First, we fitted the model:

$$Y_4 = \beta_0 + \beta_2 X_2 + \beta_3 X_3 + \beta_{23} X_2 X_3 + \epsilon .$$

The residual plot of this model appears in Figure 8. Again, observation #7 was pointed out as an outlier.



**Figure 8.** Standardized residuals versus fitted values in model (M.8).

This observation was also an outlier in all the scatter plots of  $Y_4$  versus the other responses. Because of accumulating evidence, run number 7 was excluded in the estimation of the model (M.8). The results are:

$$\hat{Y}_4 = 240 - 4.07 X_2 + 10.87 X_3 + 2.23 X_2 X_3 \quad (M.9)$$

(.60)            (.60)            (.60)

and no problems appeared in the residual plot.

## 6. Main Results

The results of the analysis are summarized in Table 3.

Coefficients for Factors

Response	Constant	$X_1$	$X_2$	$X_3$	$X_4$	$X_2 X_3$	Adjusted $R^2$
Power $Y_1$ = in Product	1.109		-.038		-.095		63.2%
Power 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%

**Table 3.** Summary of results. (Observation #7 has been excluded for estimating the model corresponding to  $Y_4$ ).

The existence of interaction between factors  $X_2$  and  $X_3$  on energy consumption indicates that those factors should be analyzed jointly as in Figure 9.

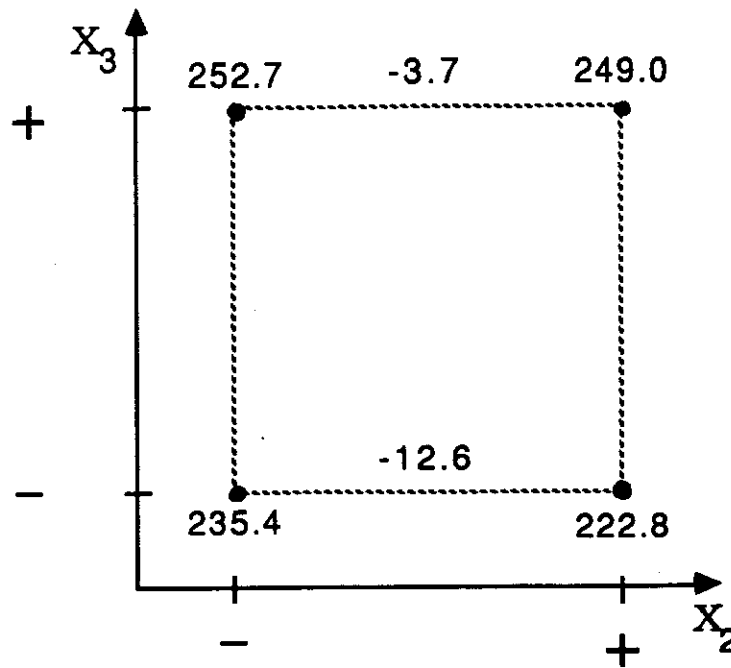


Figure 9. Mean energy consumption ( $Y_4$ ).

In order to get scientific feedback (18), the models summarized in Table 3, and especially the signs with which the different factors appear, were 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 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 in Section 3 of this paper.

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.

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 scatter plots 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 in order 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 response 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 worthwhile 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 2 1/2" 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.

## **7. 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, the number of factors and the degree of confounding of interest 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 of 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.

## **8. Acknowledgments**

We are grateful to the factory personnel that collaborated with us in the planning and running of the experiment, especially to the engineers, Mr. J. Cifuentes and Mr. X. Blanch.

### 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:

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

and we fit the model

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

then, if  $b_1 = (X_1' X_1)^{-1} X_1' y$  is the O.L.S. estimation of  $\beta_1$ , it is well known that

$$E(b_1) = \beta_1 + (X_1' X_1)^{-1} X_1' X_2 \beta_2 = \beta_1 + B \beta_2$$

where **B** matrix is the bias matrix

$$B = (X_1' X_1)^{-1} X_1' X_2 .$$

If the four main effects and the six two-factor interactions are considered as being the columns of  $X_1$ , and if  $X_2$  has as columns the four three-factor interactions and the four-factor interaction then the product  $B\beta_2$  is:

$$B\beta_2 = \begin{array}{|c|c|c|c|c|c|} \hline 0 & 0 & 0 & 0 & -1 & 123 \\ \hline 0 & 0 & 0 & 0 & -1 & 124 \\ \hline -1 & 0 & 0 & 0 & 0 & 134 \\ \hline 0 & -1 & 0 & 0 & 0 & 234 \\ \hline 0 & 0 & -0.33 & -0.33 & 0 & 1234 \\ \hline 0 & -1 & 0 & 0 & 0 & \\ \hline -1 & 0 & 0 & 0 & 0 & \\ \hline 0 & -1 & 0 & 0 & 0 & \\ \hline -1 & 0 & 0 & 0 & 0 & \\ \hline 0 & 0 & 0 & 0 & -1 & \\ \hline \end{array}$$

and therefore the confounding pattern, in the notation of (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 non-orthogonal as can be seen by computing the matrix  $(X_1'X_1)^{-1}$ . The relationship of the non-zero off-diagonal terms to the diagonal ones in this matrix is of the order: .06/.125 so that no severe non-orthogonality is present.

The matrix  $(X_1'X_1)^{-1}$  is:

$$(X_1'X_1)^{-1} = \begin{bmatrix}
 1 & 2 & 3 & 4 & 12 & 13 & 14 & 23 & 24 & 34 \\
 0.125 & .06 & \cdot & \cdot & \cdot & \cdot & \cdot & \cdot & \cdot & .06 \\
 .06 & 0.125 & \cdot & \cdot & \cdot & \cdot & \cdot & \cdot & \cdot & .06 \\
 \cdot & \cdot & 0.125 & \cdot & \cdot & \cdot & .06 & \cdot & .06 & \cdot \\
 \cdot & \cdot & \cdot & 0.125 & \cdot & .06 & \cdot & .06 & \cdot & \cdot \\
 \cdot & \cdot & \cdot & \cdot & 0.083 & \cdot & \cdot & \cdot & \cdot & \cdot \\
 \cdot & \cdot & \cdot & .06 & \cdot & 0.125 & \cdot & .06 & \cdot & \cdot \\
 \cdot & \cdot & .06 & \cdot & \cdot & \cdot & 0.125 & \cdot & .06 & \cdot \\
 \cdot & \cdot & \cdot & .06 & \cdot & .06 & \cdot & 0.125 & \cdot & \cdot \\
 \cdot & \cdot & .06 & \cdot & \cdot & \cdot & .06 & \cdot & 0.125 & \cdot \\
 .06 & .06 & \cdot & \cdot & \cdot & \cdot & \cdot & \cdot & \cdot & 0.125
 \end{bmatrix}$$

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