13.4.3 Analysis and Interpretation of Results

The data was analyzed using the `FitDefSc()` function in the `daewr` package. This function takes advantage of the special structure of the definitive screening design to find and fit an appropriate subset of the full quadratic model (10.4).

Recall that the linear main effects in definitive screening designs are independent of both the quadratic effects and linear by linear interaction effects. Thus, the `FitDefSc()` function first fits a model in the linear main effects and keeps all terms that are significant at the $\alpha = .20$ level or less. Next, forward stepwise regression steps are performed where the candidates include all quadratic and interaction terms involving the main effects in the model. This procedure sometimes results in a model with unnecessary and insignificant terms. They can be removed by backwards elimination to reach a final model.

The response data for pore diameter and the call to fit the model using the `FitDefSc()` function in the `daewr` package is shown below along with the results.

```
> pd <- c(5.35, 4.4, 12.91, 3.79, 4.15, 14.05,
+ 11.4, 4.29, 3.56, 11.4, 10.09, 5.9, 9.54,
+ 4.53,3.919,8.1,5.35)

> FitDefSc(pd,des,alpha=.05)

Call:
lm(formula = y ~ (.), data = ndesign)

Residuals:
    Min      1Q  Median      3Q     Max
-0.8140 -0.4232  0.1361  0.2253  0.8289

Coefficients:
            Estimate Std. Error t value Pr(>|t|)
(Intercept)   5.6710     0.4751  11.936 6.59e-06 ***
A             0.7664     0.2067   3.708  0.00757 **
E             0.7428     0.2067   3.594  0.00881 **
A:E          -0.5987     0.2269  -2.639  0.03349 *
B             0.4514     0.2067   2.184  0.06525 .
E:B          -0.9565     0.2439  -3.921  0.00574 **
I(A^2)        1.8801     0.5301   3.547  0.00938 **
C            -0.8758     0.2067  -4.238  0.00385 **
F             3.1508     0.2067  15.246 1.26e-06 ***
H            -0.5814     0.2067  -2.813  0.02602 *
---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1
```

```
Residual standard error: 0.7733 on 7 degrees of freedom
Multiple R-squared:  0.9797,Adjusted R-squared:  0.9536
F-statistic: 37.57 on 9 and 7 DF,  p-value: 4.187e-05
```

This model is a subset of a full quadratic model in factors A, B and E with additional linear terms involving factors C, F and H.

$$\begin{aligned}\text{Pore Diameter} = 5.67 + 0.774x_1 + 0.45x_2 - 0.88x_3 + 0.74x_5 + 3.15x_6\\ - 0.58x_8 + 1.885x_1^2 - 0.60x_1x_5 - .96x_2x_5,\end{aligned}$$

where x_1 is the coded levels of A (speed of H_2O addition), x_2 is the coded level of B (amount of H_2O), x_3 is the coded level of C (drying time), x_5 is the coded levels of E (calcination ramp), x_6 is the coded level of F (calcination temperature), and x_8 is the coded level of H (dopant amount).

Contour plots can be used to identify conditions necessary to produce TiO_2 with pore diameters useful for various applications. The R code below uses the `contour` function in the `rsm` package to make four contour plots of pore diameter with factors A (speed of H_2O addition) and E (calcination ramp) on the axes, and different fixed levels of factors B (amount of H_2O), C (drying time), F (calcination temperature), and H (dopant amount).

```
> modr<-lm(pd~A+E+A:E+B+B:E+C+F+H+I(A^2),data=des)
> library(rsm)
> par (mfrow=c(2,2))
> t<-c(-1,-1,1,-1)
> names(t)<-c("C","B","F","H")
> contour (modr, E~A, at=t, nlevels = 6,
         xlabs=c("Speed of H20 Addition (coded levels)",
         "Calcination Ramp (coded levels)"))
> t<-c(1,-1,1,-1)
> names(t)<-c("C","B","F","H")
> contour (modr, E~A, at=t, nlevels = 6,
         xlabs=c("Speed of H20 Addition (coded levels)",
         "Calcination Ramp (coded levels)"))
> t<-c(-1,1,1,-1)
> names(t)<-c("C","B","F","H")
> contour (modr, E~A, at=t, nlevels = 6,
         xlabs=c("Speed of H20 Addition (coded levels)",
         "Calcination Ramp (coded levels)"))
> t<-c(1,1,1,-1)
> names(t)<-c("C","B","F","H")
> contour (modr, E~A, at=t, nlevels = 6,
         xlabs=c("Speed of H20 Addition (coded levels)",
         "Calcination Ramp (coded levels)"))
> par (mfrow=c(1,1))
```

Figure 13.5 *Contour Plots of Pore Diameter*

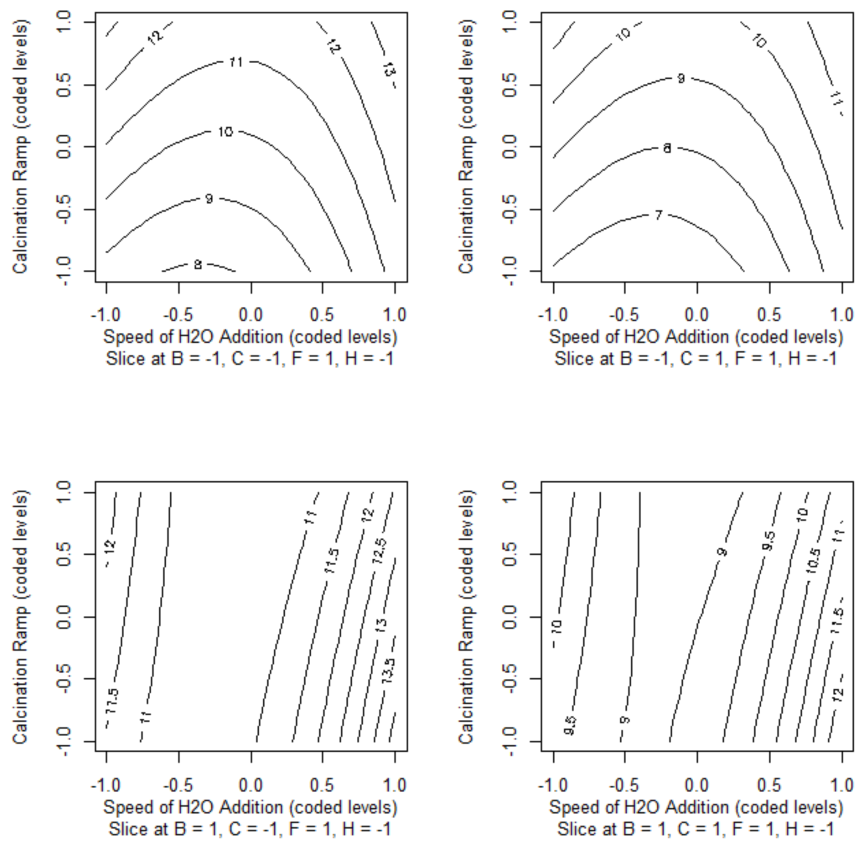


Figure 13.5 shows the four contour plots. From the plots it can be seen that the fitted equation predicts pore diameters that range from 7 to 13. Therefore, it was envisioned that TiO_2 catalyst supports for a variety of applications could be created at will. Similar equations were fit to the pore volume and the surface area responses. The fitted equations were used to predict the synthesis conditions that would result in low, medium, and large pore diameters (for various applications) while maintaining a large surface area and pore volume. Confirmation experiments verified that these predictions were accurate.

This example illustrates the progression from a preliminary exploration stage to an optimization stage of the knowledge line using just two experimental designs in sequence. The definitive screening designs allowed progression from a screening experiment with eight quantitatively leveled factors to response surface type optimization without collecting additional data.

13.5 Evolutionary Operation

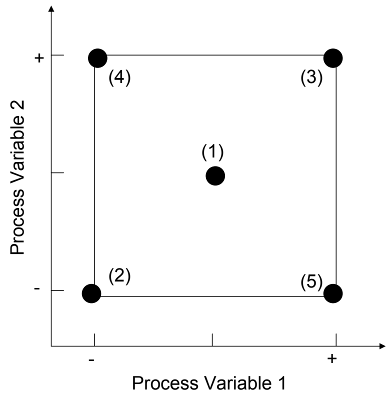
The idea of using optimization experiments, or any experimentation for that matter, is often met with resistance in an operating manufacturing facility. In manufacturing, the key process variables thought to influence production are usually tightly controlled in order to ensure the quality of the manufactured product. Tampering or experimenting with the levels of key process variables could result in the production of off-grade or scrap product. However, the levels at which the process variables are controlled in a manufacturing process may not be optimal. These levels may have been determined in pilot plant or laboratory experiments where conditions may differ in many ways from the actual manufacturing facility. In other cases, where the levels of key process variables were actually determined through preliminary experiments in the manufacturing facility, they may no longer be optimal due to drift over time in raw materials or environmental conditions. Therefore, although acceptable product is being produced with current process variable settings, there may be benefits in experimenting to obtain improved settings or to counteract drift over time.

Box (1957) proposed a method for finding improved settings of key process variables without upsetting production. He believed that a manufacturing process should not only produce product, but also information that can be used to improve the process over time. Data is normally collected in manufacturing facilities regarding (1) the settings of key process variables during each production run (to ensure operating procedures are being followed) and (2) measured characteristics of product (to monitor quality). While recording this informaton, Box suggested making small perturbations in key process variable settings from time to time according to a statistical plan like a 2^2 or 2^3 experiment. The planned changes in the key process variables would be small enough that the product output would not be degraded, yet large enough that potential process improvements could be recognized after a number of cycles through the planned settings. He called this method *Evolutionary Operation* or EVOP for short.

When using EVOP, the center point in a 2^2 or 2^3 design is assigned to the current operating conditions (in terms of what is believed to be the key process variables). Figure 13.6 illustrates the plan for two process variables. The five different process settings would each be run during one block of manufacturing. Running all five of these conditions completes one cycle of the EVOP.

If the average measure of product characteristic is y_{ijk} during the kth EVOP cycle at process variable settings i and j, then after two or more cycles, the main effect of process variable 1 $(\bar{y}_{+\cdot\cdot} - \bar{y}_{-\cdot\cdot})$, the main effect of process variable 2 $(\bar{y}_{\cdot+\cdot} - \bar{y}_{\cdot-\cdot})$, their interaction effect $((\bar{y}_{++\cdot} + \bar{y}_{--\cdot}) - (\bar{y}_{+-\cdot} + \bar{y}_{-+\cdot})$ and the curvature effect $(\bar{y}_{00\cdot} - \bar{y}_{\cdot\cdot\cdot})$ can be calculated. The standard error of the main effect and interaction estimates is $2s_p/\sqrt{4 \times r}$, and the standard error of the curvature effect is $2s_p \times \sqrt{1/r + 1/4r}$, where s_p is the pooled standard deviation over the

Figure 13.6 *A 2^2 Plan for EVOP*



five treatment combinations and r is the number of cycles of EVOP performed. The statistical significance of the effects can be determined by dividing the effect by its appropriate standard error and comparing it to the reference t-distribution with $5 \times (r-1)$ degrees of freedom. Since r is in the denominator of the standard errors, they will decrease as the number of cycles of EVOP increases.

If after five or more cycles of EVOP there is no practical or statistically significant effects, then either the process performance is optimal and stable over the experimental region, or improved operating conditions may exist outside the current experimental region. If it is believed that further improvement may be possible outside the range of the current EVOP, the ranges on the key process variables should be increased and another phase of EVOP should begin. If the effects of the key process variables (or their interaction effect) are statistically significant after two or more cycles of EVOP, a new phase of EVOP should begin where the center point of the new phase is selected to be the process condition that produced the optimum result in the last phase. If the curvature effect is statistically significant after several cycles of EVOP, and the measured product characteristics are better at the center point, the current operating conditions are optimal and EVOP should be discontinued.

Given the objective and sufficient training, EVOP should be carried out by the operators in the manufacturing facility without intervention from engineering or production management. In their book on EVOP, Box and Draper (1969) provide detailed worksheets which facilitate the EVOP calculations in a manufacturing environment. These can also be carried out simply in a modern spreadsheet program. Box and Draper suggest displaying a bulletin board in plain sight where the current EVOP status can be displayed. This will motivate the operators conducting the EVOP and inform them of the latest results.

13.6 Concluding Remarks

For each type of experimental design presented in this book, there are examples of R commands to create the design. Earlier chapters emphasized how to randomize lists of experiments to avoid bias from unknown factors and ensure valid analyses of data. In later chapters, the need for randomization was assumed, but the specific details were not emphasized since they are the same as shown in Chapter 2. In Chapter 4, the purpose for blocking experimental designs was described and the methods to create blocked designs in SAS were illustrated. The importance of blocking was emphasized throughout the remainder of the book, and situations where blocking should be used with each type of design were discussed and illustrated in the appropriate chapters.

For each type of experimental design presented, an appropriate model for the data analysis was given along with a description of the way to fit that model using R software. In Chapter 8, restricted randomization or split-plot designs were discussed and it was shown how to construct designs and analyze the data when randomization is restricted. Again the situations where this occurs and the means to handle them were emphasized in the remaining chapters of the book with all the types of designs presented. In addition, this book emphasizes the interpretation and presentation of results which is amply described and demonstrated in every chapter. The exercises at the end of each chapter provide practice for design creation and analysis and interpretation of data. They were selected to reinforce all the concepts presented in the book.

In the author's experience, the experimental design topics presented in the book cover most of the situations encountered in practice. With the power of the software in base R and the many add on packages available for R, a researcher has a powerful set of tools that simplify most research design and data analysis problems. By emphasizing where to use each of the different types of experimental designs, illustrating the creation of designs and analysis of data with R, and presenting many examples of interpretation and presentation of results, this book provides an updated guide to researchers when compared to the earlier books by Fisher (1935), Cochran and Cox (1950), and Kempthorne (1952).

After completing the study of this book, you should be able to (1) choose an experimental design that is appropriate for a research problem; (2) construct the design (including performing proper randomization and determining the proper number of replicates); (3) execute the plan (or advise a colleague to do it); (4) determine a model appropriate for the data; (5) fit the model to the data; (6) interpret the results; and (7) present them in a meaningful way to answer the research question at hand. The use of R software simplifies many of these tasks, demonstrated by the examples throughout the book.