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Introductory Design of Experiments

6.1 Introduction

Consider a chemical gas phase reaction where both pressure and temperature affect equilibrium conversion (α) so that $\alpha = \alpha\ (P,\ T)$. To study the effect of both parameters on conversion, it is customary to fix one of the two variables and vary the other until a maximum conversion is obtained then the second parameter is varied with fixing the first. This method of performing experiments suffers from a serious drawback: that is, an optimum value of one of the parameters obtained at a fixed value of the other does not necessarily represent the optimum conditions of the experiment. To illustrate this point, consider the following equation that was developed to relate equilibrium conversion of a certain reaction to P (atm) and T (K):

$$\alpha = 0.02P + .0011T - 0.95\times10^{-6} P^{0.3}.T^2 \qquad \text{(i)}$$

If pressure is kept constant, say at 1 atm., then the maximum conversion is obtained by differentiating the following equation and setting $\frac{d\alpha}{dT} = 0$

$$\alpha = 0.02 + .0011T - 0.95\times10^{-6}.T^2\ ,\ \frac{d\alpha}{dT} = 0.0011 - 2\times0.95\times10^{-6}\times T = 0$$

This yields: T = 579 K

Now, substituting T = 579 in (i), we get:

$$\alpha = 0.02P + .637 - 0.318P^{0.3} \text{ and } \frac{d\alpha}{dP} = 0.01 - 0.3\times0.318\times P^{-0.7}$$

Setting $\frac{d\alpha}{dP} = 0$, we get: P = 4.88 atm.

Actually the optimum values of P and T should have been obtained by solving the system: $\frac{\partial\alpha}{\partial T} = 0, \frac{\partial\alpha}{\partial P} = 0$. We get:

$$\frac{\partial\alpha}{\partial T} = 0.0011 - 0.95\times10^{-6}\times2\times T.P^{0.3} = 0 \qquad \text{(ii)}$$

$$\frac{\partial\alpha}{\partial P} = 0.02 - 0.95\times10^{-6}\times0.3.T^2.P^{-0.7} = 0 \qquad \text{(iii)}$$

Solving (ii) and (iii), we get: P = 3.33 atm, T = 404 K

These values are different from those obtained previously.

That is why; in the present chapter is presented a method that takes into consideration the simultaneous effect of all independent variables.

6.2 Factorial design

6.2.1 The full factorial 2^n design

A full factorial design is one in which all possible combinations of the *n* independent variables (or factors) involved in the experiment are used. Each factor can be varied along *k* levels. The number *N* of all possible combinations is:

$$N = k^n \tag{6.1}$$

For example, let in the example discussed in section 6.1, temperature be varied in steps of 50 K from 300 to 600, and pressure from 2 to 8 atm in steps of 1, then *k* = 7.

The number of experiments to be conducted to include all possible combinations of temperature and pressure = 7^2 = 49.

When the number of factors is elevated, the total number of experiments increases considerably and it is common in that case to use a two – level design in which each factor is varied only twice and equation (6.1) then becomes:

$$N = 2^n \tag{6.2}$$

Let the factors involved in the experiment be x_1, x_2, x_3, ...x_n. To each of them is assigned a lower limit $x_{i\,min}$ and an upper limit $x_{i\,max}$. The mean value of x_i over the interval ($x_{i\,min}$, $x_{i\,max}$) is termed the center of the interval:

$$\bar{x}_{0i} = \frac{x_{i\,min} + x_{i\,max}}{2} \tag{6.3}$$

And the deviation of either limit from the mean is:

$$\Delta x_i = \frac{x_{i\,max} - x_{i\,min}}{2} \tag{6.4}$$

The point with coordinates ($\bar{x}_{01}, \bar{x}_{02}, \bar{x}_{03}, \ldots, \bar{x}_{0n}$) is called the **center point of design.**

These values are changed to coded dimensionless variables z_i using the following transformation:

$$z_i = \frac{x_i - \bar{x}_{0i}}{\Delta x_i} \tag{6.5}$$

These transformations are best explained by considering the following example.

Example 6.1

The yield of a chemical reaction is affected by three factors: Temperature (T °C), pressure (P MPa) and residence time t (min.) Their upper and lower limits are respectively:100 – 200°C, 0.2 – 0.6 MPa and 10 – 30 min. The following table shows the % conversion corresponding to each set of factors:

Exp. nº	**Temp (x_1)**	**Pressure (x_2)**	**Time (x_3)**	**% Conversion**
1	100	0.2	10	2
2	200	0.2	10	6
3	100	0.6	10	4
4	200	0.6	10	8
5	100	0.2	30	10
6	200	0.2	30	18
7	100	0.6	30	8
8	200	0.6	30	12

Set the dimensionless factors table describing this experiment.

Solution:
The necessary transformations described by equations (6.3) to (6.5) have been made and the results are presented in the following table.

$$\bar{x}_1 = \frac{100+200}{2} = 150 \qquad \bar{x}_2 = \frac{0.2+0.6}{2} = 0.4 \qquad \bar{x}_3 = \frac{10+30}{2} = 20$$

$$\Delta x_1 = \frac{200-100}{2} = 50 \qquad \Delta x_1 = \frac{0.6-0.2}{2} = 0.2 \qquad \Delta x_3 = \frac{30-10}{2} = 10$$

Exp. nº	**Temp (z_1)**	**Pressure (z_2)**	**Time (z_3)**	**% Conversion (y)**
1	– 1	– 1	– 1	2
2	+ 1	– 1	– 1	6
3	– 1	+ 1	– 1	4
4	+ 1	+ 1	– 1	8
5	– 1	– 1	+ 1	10
6	+ 1	– 1	+ 1	18
7	– 1	+ 1	+ 1	8
8	+ 1	+ 1	+ 1	12

6.2.2 The design matrix

The general from of the design matrix for a 2^3 experiment takes the following form. A supplementary column is added consisting of a dummy variable $z_0 = +1$.

Exp. nº	z_0	z_1	z_2	z_3	y
1	+ 1	– 1	– 1	– 1	y_1
2	+ 1	+ 1	– 1	– 1	y_2
3	+ 1	– 1	+ 1	– 1	y_3
4	+ 1	+ 1	+ 1	– 1	y_4
5	+ 1	– 1	– 1	+ 1	y_5
6	+ 1	+ 1	– 1	+ 1	y_6
7	+ 1	– 1	+ 1	+ 1	y_7
8	+ 1	+ 1	+ 1	+ 1	y_8

This matrix satisfies the following two properties:

- The scalar product of any two column vectors = 0 (6.6)
- The sum of any column $= \sum_{i=1}^{N} z_{ji} = 0$ (6.7)

6.2.3 The regression equation

Once the design matrix is set, a regression equation is assumed in the general form:

$$y_c = a_0 + a_1.z_1 + a_2.z_2 + a_3.z_3 + a_{12}.z_1.z_2 + a_{23}.z_2.z_3 + a_{31}.z_3.z_1 + a_{123}.z_1.z_2.z_3 \qquad (6.8)$$

Where: y_c is the calculated value of y from the regression equation.

The first four terms in the above equation represent a linear model while the remaining terms are known as **interaction terms**. The determination of the 8 coefficients requires extending the design matrix to include combinations of $z_i \times z_j$ $(i \neq j)$ as well as $z_1.z_2.z_3$.

For example, the value of $z_1.z_2$ for the first set of conditions = $-1\times-1=+1$ and for the second set = $+1\times-1=-1$, so that using the data of example (6.1), the matrix shows as follows:

nº	z_0	z_1	z_2	z_3	$z_1.z_2$	$z_2.z_3$	$z_3.z_1$	$z_1 z_2 z_3$	y
1	+ 1	– 1	– 1	– 1	+ 1	+ 1	+ 1	– 1	2
2	+ 1	+ 1	– 1	– 1	– 1	+ 1	– 1	+ 1	6
3	+ 1	– 1	+ 1	– 1	– 1	– 1	+ 1	+ 1	4
4	+ 1	+ 1	+ 1	– 1	+ 1	– 1	– 1	– 1	8
5	+ 1	– 1	– 1	+ 1	+ 1	– 1	– 1	+ 1	10
6	+ 1	+ 1	– 1	+ 1	– 1	– 1	+ 1	– 1	18
7	+ 1	– 1	+ 1	+ 1	– 1	+ 1	– 1	– 1	8
8	+ 1	+ 1	+ 1	+ 1	+ 1	+ 1	+ 1	+ 1	12

The 8 coefficients can be written in form of a column vector as follows:

$$\boldsymbol{A} = \begin{vmatrix} a_0 \\ a_1 \\ a_2 \\ a_3 \\ a_{12} \\ a_{23} \\ a_{31} \\ a_{123} \end{vmatrix} \qquad \text{The dependent variable vector is: } \boldsymbol{Y} = \begin{vmatrix} 2 \\ 6 \\ 4 \\ 8 \\ 10 \\ 18 \\ 8 \\ 12 \end{vmatrix}$$

Whereas the square design matrix is:

***M* =**

+ 1	– 1	– 1	– 1	+ 1	+ 1	+ 1	– 1
+ 1	+ 1	– 1	– 1	– 1	+ 1	– 1	+ 1
+ 1	– 1	+ 1	– 1	– 1	– 1	+ 1	+ 1
+ 1	+ 1	+ 1	– 1	+ 1	– 1	– 1	– 1
+ 1	– 1	– 1	+ 1	+ 1	– 1	– 1	+ 1
+ 1	+ 1	– 1	+ 1	– 1	– 1	+ 1	– 1
+ 1	– 1	+ 1	+ 1	– 1	+ 1	– 1	– 1
+ 1	+ 1	+ 1	+ 1	+ 1	+ 1	+ 1	+ 1

In matrix form equation (6.8) reads:

$\boldsymbol{Y = M.A}$ (6.9)

Hence $\boldsymbol{A = M^{-1}.Y}$ (6.10)

Example 6.2

Find the values of the regression coefficients for the data of example (6.1)

Solution:
Using the EXCEL function MINVERSE, $\boldsymbol{M^{-1}}$ shows as follows:

0.125	0.125	0.125	0.125	0.125	0.125	0.125	0.125
-0.125	0.125	-0.125	0.125	-0.125	0.125	-0.125	0.125
-0.125	-0.125	0.125	0.125	-0.125	-0.125	0.125	0.125
-0.125	-0.125	-0.125	-0.125	0.125	0.125	0.125	0.125
0.125	-0.125	-0.125	0.125	0.125	-0.125	-0.125	0.125
0.125	0.125	-0.125	-0.125	-0.125	-0.125	0.125	0.125
0.125	-0.125	0.125	-0.125	-0.125	0.125	-0.125	0.125
-0.125	0.125	0.125	-0.125	0.125	-0.125	-0.125	0.125

Where as the column vector $\boldsymbol{Y} = \begin{vmatrix} 2 \\ 6 \\ 4 \\ 8 \\ 10 \\ 18 \\ 8 \\ 12 \end{vmatrix}$

Hence the column vector A is obtained from equation (6.10): $\boldsymbol{A} = \begin{vmatrix} 8.5 \\ 2.5 \\ -0.5 \\ 3.5 \\ -0.5 \\ -1.5 \\ 0.5 \\ -0.5 \end{vmatrix}$

Hence the coded regression equation is:

$$y_c = 8.5 + 2.5z_1 - 0.5z_2 + 3.5z_3 - 0.5z_1.z_2 - 1.5z_2.z_3 + 0.5z_3.z_1 - 0.5z_1.z_2.z_3$$

6.2.4 Testing the coefficients of the regression equation

The coefficients of the regression equation obtained are not necessary significant; that is, some of them may be eliminated without affecting the strength of correlation. This is done by performing a set of replicate tests at the design center of the experiment which in example (6.1) is (150, 0.4, 20) and determining the variance of the obtained values of *y*. Let the values of *y* obtained for three such tests be 8, 9 and 8.8. Their standard deviation *s* = 0.53

The standard deviation of the 8 coefficients is related to the standard deviation of replicates by:

$$s_a = \frac{s}{\sqrt{N}} \qquad (6.11)$$

So that in the present example: $s_a = \frac{0.53}{\sqrt{8}} = 0.187$

The significance of each coefficient is determined using the t – test by calculating each time the statistic:

$$t_{ijk} = \frac{|a_{ijk}|}{s_a} \qquad (6.12)$$

These are compared to critical values of *t* obtained from T.INV.2T function at a suitable value of α and $r - 1$ degrees of freedom. (Where, *r* is the number of replications at center of design). The tested hypothesis is:

H_0: $a_{ijk} = 0$

Rejecting H_0 means that the coefficient is significant. This will occur if

$t_{ijk} > t_{crit}$

Example 6.3

Estimate the significance of the regression coefficients for example (6.2)

Solution:

The following table shows the steps undertaken to test the 8 coefficients. At $\alpha = 0.05$, *d.f.* = 3 – 1 =2, $t_{crit} = 4.3$

a_{ijk}	8.5	2.5	-0.5	3.5	-0.5	-1.5	0.5	-0.5
t_{ijk}	46.434	13.363	2.67	18.71	2.67	8.02	2.673	-2.67
Result	reject H_0	reject H_0	accept H_0	reject H_0	accept H_0	reject H_0	accept H_0	accept H_0

Hence four coefficients are considered significant: 8.5, 2.5, 3.5 and – 1.5. The coded form of the regression equation becomes $y = 8.5 + 2.5z_1 + 3.5z_3 - 1.5z_2z_3$

Replacing the dimensionless variables (z) by the original variables using equation (6.5), we get: $z_i = \dfrac{x_i - \bar{x}_{0i}}{\Delta x_i}$

So that $z_1 = \dfrac{x_1 - 150}{50}, z_2 = \dfrac{x_2 - 0.4}{0.2}, z_3 = \dfrac{x_3 - 20}{10}$.

The regression equation becomes: $y_c = -12 + 0.05x_1 + 15x_2 + 0.65x_3 - 0.75x_2.x_3$

4.2.5 Testing the validity of the regression equation

A preliminary evaluation of the validity of the equation obtained can be made by calculating the determination coefficient. To this aim, equation (9.11) is used:

$$R^2 = \frac{\sum_{i=1}^{n}(y_c - \bar{y})^2}{\sum_{i=1}^{n}(y_i - \bar{y})^2} = \frac{166}{174} = 0.954$$

Calculations are shown next:

	y_{obs}	y_{calc}	$(y_{obs} - \bar{y})^2$	$(y_{calc} - \bar{y})^2$	$(y_{calc} - y_{obs})^2$
	2	1	42.25	56.25	1
	6	6	6.25	6.25	0
	4	4	20.25	20.25	0
	8	9	0.25	0.25	1
	10	11	2.25	6.25	1
	18	16	90.25	56.25	4
	8	8	0.25	0.25	0
	12	13	12.25	20.25	1
Mean	**8.5**		**174**	**166**	**8**

A more decisive criterion is to calculate an F – ratio defined by:

$$F_{calc} = \frac{\sum_{i=1}^{N}(y_i - y_{ci})^2}{N-c} / s^2 \qquad (6.13)$$

Where, s is the variance of replicate readings at center of design (0.28) and c the number of eliminated coefficients in the regression equation.

In the present case, $\frac{\sum_{i=1}^{N}(y_i - y_{ci})^2}{N-c} = \frac{8}{8-4}$ = 2, so that $F_{calc} = 2/0.53^2 = 7.12$

This is compared to the critical F – value obtained from FINV function at degrees of freedom: $d.f._n = N - c$ and $d.f._d = r - 1$ (Where, r is the number of replications at center of design). In the present case: $d.f._n = 8 - 4 = 4$ and $d.f._d = 3 - 1 = 2$. At a significance level $\alpha = 0.05$, the critical F – value is 19.24

Since $F_{calc.} < F_{crit.}$, then, the obtained regression equation fits the experimental data adequately.

6.3 Optimization by steepest ascent method

The regression equation obtained in the previous section has been derived over a limited experimentation range. It is usually required to use this equation to seek an extremum value to optimize the parameter y; one method often use is the steepest ascent method.

Consider the simple case where $y = f(x_1, x_2)$. The surface representing this relation is called the response surface. Fig.(6.1) shows a typical response surface exhibiting a maximum point. In the first figure, point represents the center of design point. Starting from that point, there is a particular path that would lead to the maximum point M. This path is known as the path of **steepest ascent**. Any other path like CK will not lead to this maximum.

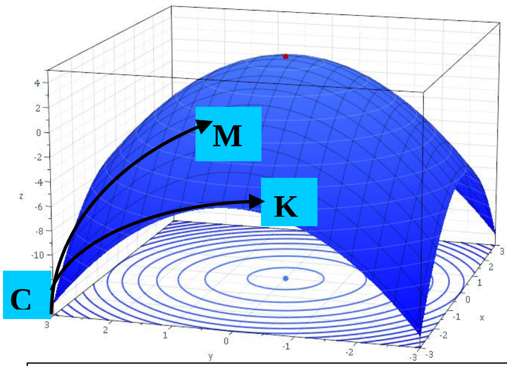

Fig 6.1: Response surface with maximum value

6.3.1 The linear model

As a start an approximate linear model can be obtained to follow the path of steepest ascent.

To understand how this path can be followed, the vector **Grad f** is defined as:

$$Grad(f) = \frac{\partial f}{\partial z_1}.\hat{i} + \frac{\partial f}{\partial z_2}.\hat{j} + \dots \frac{\partial f}{\partial z_N}.\hat{k} \qquad (6.14)$$

The line of steepest ascent is the one defined by the direction of this vector.

In **case of a linear regression model** in the form:

$$y_c = a_0 + a_1.x_1 + a_2.x_2 + a_3.x_3 + \dots$$

The partial derivatives are the coefficients of the different terms:

$$\frac{\partial f}{\partial z_1} = a_1, \frac{\partial f}{\partial z_2} = a_2, ...$$

So that: to move along this path, one chooses an increment corresponding to each variable. Let the increment corresponding to the variable z_i be Δz_i, then the corresponding increment in any other variable should be proportional to the coefficient of this variable in the regression equation. That is:

$$\frac{\Delta z_i}{\Delta z_j} = \frac{a_i}{a_j} \qquad (6.15)$$

We start at the design center (0, 0, 0, …) and choose the increment of the variable (i) which has the highest coefficient Δz_i, then we calculate the increments of the other variables using equation (6.15).

For example, let the first dimensionless variable z_1 that which has the highest coefficient (a_1). Let the chosen increment = Δz_1

Then $\Delta z_2 = \frac{a_2}{a_1}.\Delta z_1$, $\Delta z_3 = \frac{a_3}{a_1}.\Delta z_1$, etc…

Hence, $z_{11} = 0 + \Delta z_1$, $z_{21} = 0 + \Delta z_2$, $z_{31} = 0 + \Delta z_3$, … and

$z_{12} = z_{11} + \Delta z_1$, $z_{22} = z_{12} + \Delta z_2$, $z_{32} = z_{13} + \Delta z_3$, …

Generally: $z_{1,i+1} = z_{1i} + \Delta z_1$, $z_{2,i+1} = z_{1i} + \Delta z_2$, $z_{3,i+1} = z_{1i} + \Delta z_3$, …

A series of experiments is then undergone at the obtained values of z_{1i}, z_{i2}, z_{i3}, etc…for value of i = 1, 2, 3, etc. until the value of y stabilizes which means that a maximum value is obtained.

Example 6.4

Solid state sintering of magnesia is governed by soaking time (t h), temperature (T°C) and compacting pressure (p MPa). A 2^3 factorial experiment gave the following results for bulk density of pressed compacts (g/cm^3).

Four replicate experiments performed at the center of design gave the following bulk densities: 2.425, 2.415, 2.42, 2.4056.

Perform a full factorial design at significance level = 0.05 and use the steepest ascent method to show how you would reach the optimum conditions

Nº	Time	Temp	Pressure	Density
1	1	1100	20	2.37
2	5	1100	20	2.44
3	1	1500	20	2.44
4	5	1500	20	2.39
5	1	1100	40	2.41
6	5	1100	40	2.45
7	1	1500	40	2.43
8	5	1500	40	2.41

Solution:

Center of experiment: $x_1 = t$ = 3 h, $x_2 = T$ = 1300°C, $x_3 = P$ = 30 MPa

Deviations: $\Delta x_1 = 2$, $\Delta x_2 = 200$, $\Delta x_3 = 10$

The dimensionless matrix is:

Nº	Dummy	Time	Temp	Pressure	Density
1	1	-1	-1	-1	2.31
2	1	1	-1	-1	2.44
3	1	-1	1	-1	2.38
4	1	1	1	-1	2.43
5	1	-1	-1	1	2.38
6	1	1	-1	1	2.45
7	1	-1	1	1	2.42
8	1	1	1	1	2.41

The design dimensionless matrix ***M*** is:

x_0	x_1	x_2	x_3	$x_1.x_2$	$x_2.x_3$	$x_3.x_1$	$x_1.x_2.x_3$
1	-1	-1	-1	1	1	1	-1
1	1	-1	-1	-1	1	-1	1
1	-1	1	-1	-1	-1	1	1
1	1	1	-1	1	-1	-1	-1
1	-1	-1	1	1	-1	-1	1
1	1	-1	1	-1	-1	1	-1
1	-1	1	1	-1	1	-1	-1
1	1	1	1	1	1	1	1

The inverse matrix is then obtained and multiplied by the column vector corresponding to density values. This yields the coefficient column vector

$$\boldsymbol{A} = \begin{bmatrix} 2.4025 \\ 0.03 \\ 0.0075 \\ 0.0125 \\ -0.02 \\ -0.008 \\ -0.015 \\ 0 \end{bmatrix}$$

The dimensionless regression equation is then:

$$y = 2.4025 + 0.03z_1 + 0.0075z_2 + 0.0125z_3 - 0.02z_1z_2 - 0.008z_2z_3 - 0.015z_3z_1$$

The next step is to eliminate insignificant coefficients by considering the four replicate values at center of design: 2.425, 2.415, 2.41, 2.406.

Their standard deviation = 0.0082. The standard deviation of coefficients is therefore: $s_a = \frac{0.0082}{\sqrt{8}} = 0.0029$

The following table shows the steps undertaken to test the coefficients for the null hypothesis: H_0: $a_{ijk} = 0$

At $\alpha = 0.05$, $d.f. = 4 - 1 = 3$, $t_{crit} = 3.18$

a_{ijk}	2.4025	0.03	0.0075	0.0125	-0.02	-0.008	-0.015	0
t_{ijk}	828	10.34	2.58	4.31	6.89	2.58	5.17	0
Result	reject H_0	reject H_0	accept H_0	reject H_0	reject H_0	accept H_0	reject H_0	accept H_0

The final equation is;

$$y = 2.4025 + 0.03 z_1 + 0.0125 z_3 - 0.02 z_1.z_2 - 0.015 z_2.z_3 \qquad (6.16)$$

This is tested for validity using the R^2 criterion. Following equation (9.11), we get: $\boldsymbol{R^2 = 0.999}$

Also, from equation (6.13), for $N = 8$ and $c = 3$, we get:

$$\frac{\sum_{i=1}^{N}(y_i - y_{ci})^2}{N - c} = \frac{0.0009}{8 - 3} = 0.00018$$

The calculated value of $F = \frac{0.00018}{0.0082^2} = 2.67$

This is compared to the critical F – value obtained from FINV function at degrees of freedom: $d.f._n = n - c$ and $d.f._d = r - 1$. In the present case: $d.f._n = 8 - 3 = 5$ and $d.f._d = 4 - 1 = 3$. At a significance level $\alpha = 0.05$, the critical F – value is 9.013 obtained regression equation is adequate.

The actual equation can be obtained from:

$$z_1 = \frac{x_1 - 3}{2}, z_2 = \frac{x_2 - 1300}{200}, z_3 = \frac{x_3 - 30}{10}$$

This way we get:

$$y = 1.8325 + 0.08 x_1 + 0.000375 x_2 + 0.011 x_3 - 0.00005 x_1.x_2 - 0.0000075 x_2.x_3 \qquad (6.17)$$

To follow the steepest ascent direction, we arbitrarily choose an increment of time $\Delta x_1 = 0.5$ h. This corresponds to a dimensionless increment $\Delta z = 0.25$

The increment in temperature, from the obtained dimensionless model is zero. So, we can move along with a fixed temperature of 1300°C.

As for pressure, the increment is calculated from equation (6.15) to be: $\frac{0.0125}{0.03} \times 0.25 = 0.104$ (corresponding to a pressure increment = $0.104 \times 10 = 1.04$ MPa)

We have to note, however, that a linear approximation has been made as the regression equation contains a non – linear term $z_2.z_3$

The suggested experimental conditions to be followed towards maximum density will then yield the following values for density by substitution in equation (6.17):

Step nº	Time	Temp.	Pressure	ρ g.cm^{-3}
1	3	1300	30	2.373
2	3.5	1300	31.04	2.380
3	4	1300	32.08	2.388
4	4.5	1300	33.12	2.396
5	5	1300	34.16	2.404

The last entry for time is 5 hours, as this is its maximum value. Therefore, according to that model, a maximum density would be obtained at the following conditions: t = 5 h, T = 1300ºC and P = 34.16 atm. One gets a maximum value of density = 2.404 g/cm^3.

6.4 Exercise problems

(1) The strength of polystyrene boards (kPa) depends on their porosity and temperature of exposure. In a 2^2 experiment, the following design center is chosen: porosity = 0.6 with step = 0.15 and temperature = 30ºC and step = 10ºC. The following results were obtained:

Step	Porosity	Temp.	Strength
1	0.45	20	450
2	0.75	20	180
3	0.45	40	300
4	0.75	40	125

Five replicate experiments performed at the center of design gave the following values of strength: 256, 284, 270, 278, 250 kPa

Perform a full factorial design at significance level = 0.025 and use the steepest ascent method to show how you would reach the optimum conditions.

(2) To study the effect of temperature, time and particle size on the extraction of a certain liquid from a solid a full factorial two – level design was performed. The results are listed in the following table:

The last three rows represent a replicate at the center of the design.

Derive a first order regression equation showing the dependence of yield on the three factors then check the significance of the different coefficients.

Also check the validity of the system. (Take α = 0.05)

Time	Temp	Particle size	Yield
20	40	0.1	16
20	40	0.3	14
20	80	0.1	21

20	80	0.3	17
40	40	0.1	23
40	40	0.3	21
40	80	0.1	26
40	80	0.3	24
30	60	0.2	21
30	60	0.2	22
30	60	0.2	23

(3) The following data have been obtained on studying the effect of three variables: temperature of calcination (ºC), time of leaching (h) and acid concentration (%) on the concentration of aluminum sulfate in solution upon treatment of kaolin with sulfuric acid. Four specimens investigated at the center of design (750ºC, 3 h, 40%) yielded concentrations of 6.82%, 7.27%, 6.77% and 7.8%. Derive a regression equation showing the dependence of yield on the three factors then check the significance of the different coefficients.

Also check the validity of the system. (Take $\alpha = 0.05$)

#	Temp. ºC	Time h	Conc. %	Yield %
1	600	2	20	8.27
2	600	2	60	7.08
3	600	4	20	7.36
4	600	4	60	9.31
5	900	2	20	4.95
6	900	2	60	4.45
7	900	4	20	6.22
8	900	4	60	2.57