THE STRATEGY OF EMPIRICAL RESEARCH AND OPTIMIZATION PROCESS

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Abstract

Empirical modeling is considered from the perspectives of a general scheme for the Strategy of Empirical Research and Optimization Process (SEROP). This approach intends to facilitate the understanding of the necessary steps to arrive to mathematical models able to appropriately describe the behavior of a group of controllable independent variables related to a certain response. Aspects connected with definition of the problem, variable’s identification and optimization stages are discussed. As an example of SEROP application, it is presented the empirical modeling of the basic extraction of alginic acid from brown algae.

Keywords: empirical optimization, empirical research, optimization strategy.
1. Introduction

The study of a technical or scientific situation, usually presents two different and, in many ways, complementary points of view. These are:

a) a generalized phenomenological description and;

b) a limited empirical modelling.

Human knowledge is continuously fed by both approaches and there are countless examples of interactions between them. When the problem is to find a proper description of a process in a short time, at low costs and with the necessary accuracy, the dilemma emerges. Although theoretical models are to be preferred, they are unfortunately not available for every new practical situation. In these cases, empirical models may be used, knowing that their results are limited to the experimental region in which they were obtained. This drawback can be of no importance if what is sought is the behavior of a particular process in a set of particular conditions. However, empirical models building has also its own rules, that should be followed in order to arrive at reliable information.

Author’s own experience in the field of empirical modelling is presented in this paper as a modest contribution for better planned and interpreted experimental work.

The paper is divided in three parts:

1. the search for the optimum;
2. practical considerations;
3. illustrative example.

2. The search for the optimum

In a well defined problem, where responses are correctly selected and where a screening process for the reduction of the identified independent variables has been applied, a search for the optimum can be conducted. The search is limited to an experimental region in which only one stationary point should be found.

Figure 1 shows the hypothetical boundaries of an experimental region where responses are plotted as contour lines, considered factors are \( X_1 \) and \( X_2 \). Points I and II represented the zones where initial exploring experimental designs are carried out and Point C stands for the optimum (a maximum in this example).

The search is initiated somewhere inside the experimental region and each new experiment is conditioned by the obtained response of the previous one. Vector I-II shows the direction to follow until a significant decrease in response is found. Then, a readjustment of variable’s levels is done in a way as to get increasing responses through the new design.
The procedure is repeated until curvature effects begin to be noticeable. In this point of the search, it is advisable to establish the trajectory for best performance by means of the steepest ascent method.

At the end of this last step, a new two level factorial design is conducted. The resulting model will show a big curvature effect, indicating that a second order design will describe the exact position of the stationary point. The obtained second order model is best analyzed through its response surface.

The search for the optimum is in no way a simple trial and error process and needs careful attention in every step. At this point it should be remembered that not all empirical studies have to end with an optimization process. This case takes place when the only objective is to describe conditions under which a certain process operates and there is no intention or possibility to change it.

In order to organize the different stages in the search for the optimum and also to include the simpler situation just mentioned above, a general scheme for the strategy of empirical research and optimization process (SEROP) is presented in Figure 2.

### 3. Practical considerations about steps of SEROP

**Problem definition:** To define the objective of an investigation, it is necessary to conduct an information searching process that will allow answering the following questions:

- **Which will be the responses to be studied and which is their hierarchy order?**
  
  It is important to establish a hierarchical order when more than one answer is selected, because they will be affected by the same factors, making it frequently impossible to find a combination of the given factors that optimizes all answers at the same time.

- **Which are the independent factors that may affect the answers?**
  
  The larger the number of independent factors, the larger the number of experiments. The efficiency in this step will be strongly related to the investigator’s selection capacity.
Definition of the problem:
a) Objectives definition  
b) Selection of responses according to defined objectives

Identification of involved independent variables (factors) and their levels

Factor’s screening experimental designs up to finding k=3 of 4 most important factors

Formulation of a $2^k$ full factorial design ($k \leq 4$)

Is there pronounced curvature in the selected experimental zone?

What is it wished?

To find an optimum

To study factor’s behavior in a selected experimental zone

Conduct additional experiments until curvature is detected

Follow the steepest ascent trajectory

Apply lack of fit test and validate final model

Apply Haaland’s strategy

Adjust a second order model

Make response surface analysis

Determine optimum coordinates and make comprobatory experiments in it’s neighborhood

End

Figure 2 – Scheme showing the Strategy of Empirical Research and Optimization Process (SEROP)
3.1 Selection of the experimental zone under study

The space occupied by the experiment will be determined by the separation that exists between the higher and lower levels of each factor.

1. if the levels are chosen too closely, it is possible that the response variation (Y) is not observed in the experiment or may have the order of magnitude of the stochastic fluctuations, and therefore the model will be \( Y = \text{constant} \). (Figure 3, \( a \leq X_i \leq b \))

2. if the levels are chosen too far apart, two mistakes may be made. (Figure 3, \( a \leq X_i \leq d \))
   - The points “a” and “d” may be found on both sides of a maximum or a minimum and the difference between the Y value results may be impossible to be observed.
   - The experimental errors made at different points of the region being significantly distinct, indicating in this case a lack of variance homogeneity and therefore the utilized parametric tests, for example t and F, would not be powerful enough, because they are based on the assumption that variance homogeneity exists, among other conditions.

The interval (Figure 3, \( a \leq X_i \leq c \)) gives more reliable results, since it contains relevant changes in the response. Naturally, before conducting the experiments, these situations are unknown, therefore the initial selection of values of the operating variables will be affected by Guerra Debén & Sevilla (1988):

a) the historical knowledge of the system under study;
b) the available theory;
c) the existence of exploratory experiments;
d) the luck factor.

![Figure 3](image)

**Figure 3** – Behavior of response Y when the level of the X_i factor is varied

3.2 The screening of independent variables (Sutton, 1997; Box *et al.*, 1993; and Barros Neto *et al.*, 1995)

To avoid selecting a factor that carries little or no significance use should be made of known screening techniques (Sutton, 1997; Box *et al.*, 1993; and Barros Neto *et al.*, 1995). It should be borne in mind that initially identified factors must be reduced in number in order to follow an optimization strategy.
If a factor of strong influence over the response, is not controlled, it won’t be possible to obtain reliable results due to the large fluctuations that are to be observed in the response, and what is even worse, the noted behavior can be wrongly attributed to some of the controlled factors. It can also be the case that it is not possible to introduce changes in a known important factor. In such cases, the wisest thing to do is to control it at a definite favorable level. Thus, the obtained results are said to be conditioned or restricted by the factor.

3.3 Formulation of a $2^k$ full factorial design

When screening work is finished, the number of independent variables should be around 4 or less. It is possible, however, to count with a fractioned design having also a maximum of 16 experiments in its planning. These numbers are not rigid limits and specific conditions will have the last word. Box et al. (1993) as well as Barros Neto et al. (1995) are excellent treatises in this respect.

3.4 Curvature check

When a supposed linear model, contains interaction’s coefficients as big as or even bigger than individual factor’s coefficients, the corresponding response surface will show curvature. There are cases where curvature, although present, is rather slight and the obtained model shows no lack of fit. However, if curvature is pronounced, linear models are not able to represent experimental behavior and need to be replaced by second order models.

Curvature can also be evaluated quantitatively by means of central points replicates. This will be shown in the illustrative example.

3.5 Establishing the trajectory for best performance

If the main objective of the research is to find either a maximum or a minimum for the studied response, several strategies are available. One, or perhaps the most known method, is the method of steepest ascent (Box et al., 1993), which makes use of a few exploratory points conducted according to a stepwise change in the levels of the involved factors. This change is determined by a reference factor, that usually is the one with the highest coefficient in the linear model.

In this way, an experimental trajectory is followed until a change is noted in the direction of response’s increase. This point of rupture gives the necessary information about both sides of the noted change (possible stationary point) and allows a proper factor levels selection for a new two level factorial design. The resulting linear model will show a great curvature effect. This last obtained linear model is used at the nucleus of a second order design, which requires additional experimental points to fully describe the optimum zone.

If the search for the optimum is, by any reason, initiated very close to the stationary point, models with significant curvature effects will be obtained and the steepest ascent method looses efficiency. In those cases is better to apply the techniques recommended by Haaland (1989).
3.6 Second order design (Montgomery, 1991)

Two of the most used second order designs are:

- Central composite orthogonal designs;
- Central composite rotational designs.

Both types of designs are carried out sequentially. This means that the information obtained from a full or fractional design is used in the data processing after adding new experiments according to the characteristics of each design.

3.6.1 Central composite orthogonal designs (CCOD)

These designs are distinguished by (see Table 1):

- having a nucleus made out of a full factorial design or a fraction of it;
- two extra runs for each factor, located at a coded $\alpha$ distance from the center of the experimental region. These are called axial points as they are at (+) $\alpha$ and (–) $\alpha$ distance from the zero point on the factors axes.
- one or more runs at the center of the design.

| Number of Factors | Design’s Nucleus (fraction of a full $2^k$ design) | Number of Runs | Total Runs | $\alpha$ distance for axial points |
|-------------------|--------------------------------------------------|----------------|------------|----------------------------------|
|                   | at the nucleus                                   | at the axis    | at the center |                                  |
| 2                 | 1                                                | 4              | 4           | 1                                 |
| 3                 | 1                                                | 8              | 6           | 1                                 | 15                      | 1.215                  |
| 4                 | 1                                                | 16             | 8           | 1                                 | 25                      | 1.414                  |
| 5                 | $\frac{1}{2}$                                    | 16             | 10          | 1                                 | 27                      | 1.547                  |

Second order CCOD have higher precision when:

- optimum finds itself in the close neighborhood of experimental zone’s center. This is perfectly possible if an adequate optimization strategy (i.e. – steepest ascent method) has been followed or the investigators own experience indicates this is so;
- there are no time changes in experimental responses between first experiments (at the nucleus) and additional experiments (center and axis);
- variance is approximately the same for all experimental points located at equal distance from the center of the design.

3.6.2 Central composite rotational designs (CCRD)

This type of second order design assures the same standard error in predicted values for all experimental points located at the equal distance from the center of the design. Usually the precise nature of the response surface is not known before experimental work begins.

In this situation, it is highly improbable to distribute planned experimental points along maximum (minimum) surface slope in a way as having also minimum variance. This is why CCRD are very appreciated. Table 2 shows the structure of these designs.
Table 2 – Structure of CCRD

| Number of Factors | Design’s nucleus (fraction’s of a full 2^K design) | Number of Runs | Total Runs | α distance for axial points |
|-------------------|-----------------------------------------------|----------------|------------|--------------------------|
|                   | at the nucleus | at the axis | at the center |                      |
| 2                 | 1              | 4          | 4           | 5                       | 13                       | 1.414                      |
| 3                 | 1              | 8          | 6           | 6                       | 20                       | 1.682                      |
| 4                 | 1              | 16         | 8           | 7                       | 31                       | 2.000                      |
| 5                 | 1/2            | 16         | 10          | 6                       | 32                       | 2.000                      |

4. Example of SEROP

Alkaline extraction of alginic acid from brown algae (Mesa Pérez et al., 1998)

Problem Definition: Brown algae, previously crushed and acidified with HCl solution, contains alginic acid that is intended to be extracted in the form of its sodium salt.

The final product, once purified, has many industrial applications based on the viscosity of its aqueous solutions. The research objective is to test an extraction method based on the reaction of a solution of sodium carbonate with alginic acid inside acid treated algae residues. The operation is batch wise conducted at room temperature (26ºC) under agitation.

To evaluate if the objective is achieved, two responses are to be followed:

- yield of sodium alginate;
- viscosity of 1% aqueous solution of sodium alginate.

To be brief, only viscosity will be treated here.

4.1 Identification of factors

Initial acid treated algae as well as sodium carbonate solutions came from the same stock. All experiments were performed in the same reactor with the same reaction time and same operators.

A constant agitation speed was adopted throughout all experimental work. Under these conditions no screening was considered necessary and the following factors were identified together with their levels (see Table 3). Of course, this is a simplified situation. Screening work is almost always necessary when beginning to study a new problem.

Table 3 – Alkaline extraction of acid treated brown algae (identified factors, symbols, and levels)

| Factor                        | Coded Symbol | Levels (coded) |
|-------------------------------|--------------|----------------|
|                               |              | Inferior (−1) | Central (0) | Superior (+1) |
| Temperature (ºC)              | X_1          | 30             | 45           | 60           |
| Na_2CO_3 mass concentration (g/L) | X_2        | 0.6            | 1            | 1.4          |
| Liquid-solid ratio (kg/kg)    | X_3          | 10             | 15           | 20           |

Factor’s levels were chosen according to literature information and researcher’s own experience.
4.2 Formulation of a $2^K$ full factorial design

As $K=3$, a $2^3 = 8$ experiments full factorial design is chosen. Independent variable matrix ($X$ matrix) (includes experiment matrix), as well as response matrix ($P$ matrix) are shown below:

$$
X = \begin{bmatrix}
X_0 & X_1 & X_2 & X_3 & X_1X_2 & X_1X_3 & X_2X_3 & X_1X_2X_3 \\
1 & -1 & -1 & -1 & +1 & +1 & -1 & 1 \\
1 & +1 & -1 & -1 & -1 & +1 & +1 & 1 \\
1 & -1 & +1 & -1 & -1 & -1 & +1 & 1 \\
1 & +1 & +1 & -1 & +1 & -1 & -1 & 1 \\
1 & -1 & -1 & +1 & +1 & -1 & +1 & 1 \\
1 & +1 & -1 & +1 & +1 & -1 & +1 & 1 \\
1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\
1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\
\end{bmatrix}
$$

Following a well known procedure (Montgomery, 1991), first order model is obtained. The corresponding analysis of variance is shown in Table 4.

Table 4 – ANOVA for first order model

| Variation source | SS    | df | MSS    | F test | p-value |
|------------------|-------|----|--------|--------|---------|
| $X_1$            | 69938 | 1  | 69938  | 1427   | 0.0007  |
| $X_2$            | 29040.5 | 1  | 29040.5 | 593    | 0.0017  |
| $X_3$            | 381938 | 1  | 381938 | 7795   | 0.0001  |
| $X_1X_2$         | 6612.5 | 1  | 6612.5 | 135    | 0.0073  |
| $X_1X_3$         | 9248  | 1  | 9248   | 189    | 0.0053  |
| $X_2X_3$         | 22684.5 | 1  | 22684.5 | 463    | 0.0022  |
| Lack of fit      | 398745.4 | 2  | 199372.7 | 4069  | 0.0002  |
| Pure error       | 98    | 2  | 49     |        |         |
| Total (Corr)     | 918304.9 | 10 |        |        |         |

$R^2 = 0.57$

After testing for coefficients significance at $\alpha = 0.05$ level, the coded final model is:

$$
\mu = 656 - 93X_1 + 60X_2 - 218X_3 + 29X_1X_2 - 34X_1X_3 - 53X_1X_2X_3 \quad \ldots \quad (4.2.1)
$$

A brief look to the relative magnitude of interactions coefficients, gives a clear evidence of pronounced curvature. As it can be observed, all interactions have values of the same order
of individual factors. An analysis of variance, in this case, will show that the regression sum of squares is quite smaller than the total sum of squares and that there exists a large and, therefore, significative lack of fit in the model.

Of course, a curvature check is always possible. Thus,

\[
\text{Curvature Effect} = \left( \bar{y}_2 - \bar{y}_1 \right) \pm t_{(a,v)} S_{\exp} \tag{4.2.2}
\]

\[
\bar{y}_1 = \text{average of the } n_1 \text{ experimental responses of the design (here } n_1 = 8 \text{ and } \bar{y}_1 = 539.5) \\
\bar{y}_2 = \text{average of the } n_2 \text{ center points (here } n_2 = 3 \text{ and } \bar{y}_2 = 967) \\
t = \text{student statistic (two tailed’s test)} \\
\alpha = \text{significance level} = 0.05 \\
v = \text{degrees of freedom of } S_{\exp} \text{ (here } v = 2) \\
S_{\exp} = \text{standard deviation of pure error (here } S = 7), \text{ and so} \\
\text{Curvature Effect} = (967 - 539.5) \pm t_{(0.05, 2)}(7) = 427.5 \pm 4.3 \cdot (7) = 427 \pm 30
\]

This result shows that responses at the center points are well higher than those belonging to the base design points and constitute a quantitative proof of curvature.

A closer look to the obtained model shows that factor X_3 (liquid-solid ratio) should work at the lowest level in order to obtain greater viscosities. Figure 4, shows response surface for a fixed X_3 = –1 level. It is clearly seen, that viscosity reaches a so called saddle point, that can’t be described completely by the so far obtained model.

![Figure 4](image-url) – Response Surface for Viscosity Behavior at X3 = –1 (First order model)

On the other hand, if X_3 is held constant at +1 level Figure 5 shows a different situation, in which a plane with some curvature can be appreciated.
A tridimensional picture of both cases is given in Figure 6 and 7. In this particular case, a lowering of the liquid-solid ratio ($X_3$) should have its limits because otherwise power increase due to stirring could be too high. Also, a point may be attained where very little or no liquid exits. This is, of course, a hypothetical situation, but helps to make a reasonable appreciation of how to change factor’s levels.

**Figure 5** – Response Surface for Viscosity Behavior at $X_3 = +1$ (First order model)

**Figure 6** – Tridimensional picture of first order model for $X_3 = -1$
According to the SEROP scheme, it is recommended now to adjust a second order model due to the fact that the lineal model does not describe adequately viscosity behavior.

4.3 Adjustment of a second order model (Montgomery, 1991)

As there were evidences of a maximum in the vicinity of the initially studied experimental region, a CCOD was tried, making use of former $2^3$ full factorial as nucleus of the new design and performing 6 new experiments for the axial points ($\alpha = 1.215$). An additional center point is also added in order to improve $S_{exp}$ precision, although three of them are already done. The corresponding experimental matrix as well as the response matrix are:
After obtaining a second order model, ANOVA is conducted (Table 5).

Table 5 – ANOVA for second order model

| Variation source | SS   | df | MSS   | F test | p-value |
|------------------|------|----|-------|--------|---------|
| X₁               | 93894| 1  | 93894 | 2253   | 0.0000  |
| X₂               | 38105| 1  | 38105 | 915    | 0.0001  |
| X₃               | 525577| 1  | 525577| 12614  | 0.0000  |
| X₁ X₂            | 6613 | 1  | 6613  | 159    | 0.0011  |
| X₁ X₃            | 9248 | 1  | 9248  | 222    | 0.0007  |
| X₁ X₂ X₃         | 22685| 1  | 22685 | 544    | 0.0002  |
| X₁²              | 677441| 1  | 677441| 16259  | 0.0006  |
| X₃²              | 544965| 1  | 544965| 13079  | 0.0000  |
| X₂²              | 349098| 1  | 349098| 8378   | 0.0000  |
| Lack of fit      | 87   | 5  | 17    | 0.42   | 0.8164  |
| Pure error       | 125  | 3  | 42    |        |         |
| Total (Corr)     | 2259454| 17 |       |        |         |

R² = 0.99
R² (adjusted df) = 0.99
The final coded model, after significance tests for the coefficients and a lack of fit test for the model are done, is:

\[ \mu = 966 - 93X_1 + 59X_2 - 219X_3 + 29X_1X_2 - 34X_1X_3 - 53X_1X_2X_3 - 361X_1^2 - 324X_2^2 + 259X_3^2 \]

\[(4.3.1)\]

4.4 Analysis of response’s surfaces

If in former model \( X_3 \) is fixed at –1 level, following model is obtained:

Model 1: (\( X_3 = -1 \))

\[ \mu = 1444 - 59X_1 + 59X_2 + 782X_1X_2 - 361X_1^2 - 324X_2^2 \] \[(4.4.1)\]

![Figure 8 – Response Surface for Viscosity Behavior (Second Order model for \( X_3 = -1 \))](image)

Now, holding \( X_3 \) at +1 level, a new model can be shown:

Model 2: When \( X_3 = +1 \)

\[ \mu = 1006 - 1267X_1 + 59X_2 - 24X_1X_2 - 361X_1^2 - 324X_2^2 \] \[(4.4.2)\]
Response’s surfaces for both models are presented in Figure 7.
Both maximum points are conditioned by the values assigned to \( X_3 \) and as expected are quite different.

### 4.5 Determination of the optimum point

In order to obtain the coordinates of the stationary point \( \frac{\partial \mu}{\partial X_i} = 0 \) (\( i \) being 1 and 2) are determined for each model and from the solution of both equation systems, the conditional maximum coordinates are obtained. Thus, for model 1 (\( X_3 = -1 \)), maximum viscosity is reached at:

- \( X_1 \): Temperature = 44°C
- \( X_2 \): Na\(_2\)CO\(_3\) mass concentration = 1.036 g/L
- \( X_3 \): Liquid-solid mass ratio = 10 kg/kg
- \( \mu_{opt} \) = Maximum viscosity = 1449 mPa.s

For model 2 (\( X_3 = +1 \)), optimum conditions are given when:

- \( X_1 \): Temperature = 42°C
- \( X_2 \): Na\(_2\)CO\(_3\) mass concentration = 1.04 g/L
- \( X_3 \): Liquid-solid mass ratio = 20 kg/kg
- \( \mu_{opt} \) = 1015 mPa.s

Due to practical reasons (values of \( X_3 \) below 10 kg/kg were not recommended), model 1 was taken as the final model.
5. Conclusions

The SEROP is composed of a series of steps applicable to experimental regions in which only one stationary or optimum point exists and where all the involved factors or independent variables can be controlled. The number of necessary steps to reach the stationary point will depend in a great measure on the previous knowledge of the problem. The more knowledge of the nature of the investigated process, the less stages will have to be carried out. The presented general scheme shows a common sense way of dealing with these situations, looking for faster, cheaper and rigorous experimental procedures of research, based on an empirical approach.

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Nomenclature

CCOD Central composed orthogonal designs
CCRD Central composed rotational designs
SEROP Strategy of empirical research and optimization process

k Number of factors

\( n_1 \) Number of experimental responses of the fractional design

\( n_2 \) Number of responses at the center of the design

\( S^2_{\text{exp}} \) Pure error

\( t \) Student statistic

\( X_i \) Factor or independent variable i

\([X]\) Independent variable matrix

\( y_{\bar{1}} \) Average of \( n_1 \) responses

\( y_{\bar{2}} \) Average of \( n_2 \) responses

\( \alpha \) Distance from the zero point of a factor’s axis in CCOD and CCRD. Also significance level

\( \nu \) Degrees of freedom of \( S^2_{\text{exp}} \)

\( \mu \) Viscosity (mPa.s)

\([\mu]\) Responses matrix for viscosity

\( \frac{\partial \mu}{\partial X_i} \) Partial derivative of \( \mu \) in respect to \( X_i \)

SS Sum of squares

MSS Medium sum of squares

F -test-Fischer test

df Degrees of freedom
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