Study of the Fastest Rate of Freezing Saline Solution Using Factorial Design Method

Nia Nuraeni Suryaman

Department of Mechanical Engineering, Widyatama University, Indonesia

Abstract
An engineer needed the ability to design an experiment research to be effective and efficient to obtain optimal results. The purpose of this experiment is to determine the fastest rate of freezing saline solution. The research process begins with determining the independent variables as much as possible and determines the three independent variables to be tested. After determine variables, and then create table factorial design to determine the research steps as much as 8 times. Then determine the most influential variables using Yates's algorithm was then tested again using response surface methodology (RSM), but for this study only uses two steps of the three step RSM. So it can be concluded that the lower temperature and salinity the faster the rate of freezing for both type of salt, Krosok and salt.

Keywords Rate of Freezing, Saline Solution, Factorial Design

1. Introduction
An engineer needed the ability to design an experiment research to be effective and efficient to obtain optimal result. These experiments use factorial design methods to make it easier for researchers to get effective and efficient results.

Factorial design is a method for determining the influence of several independent variables on the response. Basically an experiment is designed to determine one variable in one response. Therefore, factorial design can make it easier to experiment with more than one independent variable. Another use of factorial design is that it can reduce the number of experiments we have to do by studying several factors simultaneously.

The purpose of this experiment is to determine the fastest rate of freezing saline solution. The output to be produce of this study is a graph and the most influential variable as a result. In this study the output will be obtained by using factorial design and least square and response surface methodology.

2. Literature Study
2.1. Factorial Design
Factorial design is an important method to determine the effects of multiple variables on a response/output. Traditionally, experiments are designed to determine the effect of one variable upon one response/output.

There are advantages by combining the study of multiple variables in the same factorial experiment. Factorial design can reduce the number of experiments one has to perform by studying multiple factors simultaneously. Additionally, it can be used to find both main effects (from each independent factor) and interaction effects (when both factors must be used to explain the outcome).

Factorial design is a useful method to design experiments in both laboratory and industrial settings. Because factorial design can lead to a large number of trials, which can become expensive and time-consuming, factorial design is best used for a small number of variables with few states (1 to 3).
2.2. Least Square

The method of least squares is a standard approach to the approximate solution of over determined systems, i.e., sets of equations in which there are more equations than unknowns.

Least square means that the overall solution minimizes the sum of the squares of the errors made in the results of every single equation. The most important application is in data fitting.

In this equation the \( \beta \)'s are unknown constants to be estimated and the \( x \) have known values. One common example is where \( x_1, x_2, \ldots \) are the levels of \( k \) factors, say temperature, line speed, concentration, and so on, and \( y \) is a measured response such as yield.

\[
y = \beta_0 x + \beta_1 x_1 + \beta_2 x_2 + \ldots + \beta_k x_k + \epsilon
\]

Table 1 shows a small illustrative set of data from an experiment to determine how the initial rate of formation of an undesirable impurity \( y \) depended on two factors: A) the concentration \( x_0 \) of monomer and B) the concentration of dimer \( x_1 \). The mean rate of formation \( y \) was zero when both components \( x_0 \) and \( x_1 \) were zero. Over the relevant ranges of \( x_0 \) and \( x_1 \) the relationship was expected to be approximated by

\[
y = \beta_0 x_0 + \beta_1 x_1 + \epsilon
\]

For any particular set of trial values of the parameters \( \beta_0 \) and \( \beta_1 \) one could calculate \( S(\beta) \). For example, for data of Table 1, if \( \beta_0 = 1 \) and \( \beta_1 = 7 \), would get:

\[
S(1,7) = \sum (y - 1x_0 - 7x_1)^2 = 1.9022
\]

Thus in principle could obtain the minimum value of \( S \) by repeated calculation for a grid of trial values. It would eventually be able to construct Figure 2, a 3D plot of the sum of squares surface of \( S(\beta) \) versus \( \beta_0 \) and \( \beta_1 \). The coordinates of the minimum value of this surface are the desired least square estimates

\[
b_0 = 1.21 \quad \text{and} \quad b_1 = 7.12
\]

\[
y = 1.21 x_0 + 7.21 x_1
\]
Table 1. Initial Rate of Impurity Investigation

| Observed Run Number | Order in Which Experiments Were Performed | Concentration of Monomer, \( x_0 \) | Concentration of Dimmer, \( x_1 \) | Initial Rate of Formation of Impurity \( y \) |
|---------------------|------------------------------------------|-----------------|-----------------|----------------------------------|
| 1                   | 3                                        | 0.34            | 0.73            | 5.75                             |
| 2                   | 6                                        | 0.34            | 0.73            | 4.79                             |
| 3                   | 1                                        | 0.58            | 0.69            | 5.44                             |
| 4                   | 4                                        | 1.26            | 0.97            | 9.09                             |
| 5                   | 2                                        | 1.26            | 0.97            | 8.59                             |
| 6                   | 5                                        | 1.82            | 0.46            | 5.09                             |

Quantities needed in subsequent calculations: \( \sum x_0^2 = 7.0552, \sum x_1^2 = 3.6353, \sum x_0 x_1 = 4.1782, \sum x_0 y = 38.2794, \sum x_1 y = 30.9388, \sum y^2 = 267.9245 \)

2.3. Normal Equation

\[
\frac{\partial S(\beta)}{\partial \beta_0} = -2(\Sigma y - \beta_0 x_0 - \beta_1 x_1) x_0 = 0
\]

\[
\frac{\partial S(\beta)}{\partial \beta_1} = -2(\Sigma y - \beta_0 x_0 - \beta_1 x_1) x_1 = 0
\]

After simplification these become what are called the normal equations

\[
b_0 \Sigma x_0^2 + b_1 \Sigma x_0 x_1 = \Sigma y x_0
\]

\[
b_0 \Sigma x_0 x_1 + b_1 \Sigma x_1^2 = \Sigma y x_1
\]

3. Research Methodology

1) Determined Independent Variable
   The independent variables determined for the design of this experiment are as follows:
   a) Freezer Temperature
   b) Refrigerator
   c) Salinity
   d) Frozen water mass
   e) Freezing time
   f) Initial water temperature
   g) Type of water
   h) Type of salt
2) After determining the independent variable for the experiment, the next step is to determine the three independent variables to be tested, while the other independent variables are fixed.

a) The independent variables to be tested are:
   - Freezer Temperature (Freezer Temperature Setting)
   - Freezer temperature settings are varied in:
     - Setting 4, with a temperature of -17°C
     - Setting 6, with a temperature of -19°C
   - Salinity
     Salinity is varied in:
     - 10% salt
     - 20% salt
   - Type of Salt
     Type of salt is varied in:
     - Salt

b) Fixed free variables are:
   - Refrigerator Brand
     Toshiba
   - Frozen Water Mass
     250 grams
   - Freezing Time
     5 hours
   - Initial Water Temperature
     25°C
   - Type of Water
     Tap water

3) Factorial Design Method
   This experiment used the $2^3$ factorial design methods with two quantitative factors, temperature and salinity and one qualitative factor, the type of salt.

| Table 2. Factorial Design $2^3$ |
|----------------------------------|
| Freezer Temperature | Salinity (%) | Type of Salt |
| + 6 (-19°C) | 20 | Krosok |
| - 4 (-17°C) | 10 | Salt |

| Table 3. Factorial Design |
|---------------------------|
| Run | Freezer Temperature (°C) | Salinity (%) | Type of Salt | Ice Mass (g) | Freezing Rate (g/jam) |
| 1 | -19 | 20 | Krosok | 30 | 6 |
| 2 | -17 | 20 | Krosok | 0 | 0 |
| 3 | -19 | 10 | Krosok | 210 | 42 |
| 4 | -17 | 10 | Krosok | 150 | 30 |
| 5 | -19 | 20 | Salt | 0 | 0 |
| 6 | -17 | 20 | Salt | 0 | 0 |
| 7 | -19 | 10 | Salt | 160 | 32 |
| 8 | -17 | 10 | Salt | 170 | 34 |

4. Results and Discussion

1) The experiment was carried out in 8x according to the design factorial table that was made before.

| Table 4. Experiment Result |
|-----------------------------|
| Freezer Temperature (°C) | Salinity (%) | Type of Salt | Ice Mass (g) | Freezing Rate (g/jam) |
| -19 | 20 | Krosok | 30 | 6 |
| -17 | 20 | Krosok | 0 | 0 |
| -19 | 10 | Krosok | 210 | 42 |
| -17 | 10 | Krosok | 150 | 30 |
| -19 | 20 | Salt | 0 | 0 |
| -17 | 20 | Salt | 0 | 0 |
| -19 | 10 | Salt | 160 | 32 |
| -17 | 10 | Salt | 170 | 34 |

2) The Most Influential Variables
   The next step after getting the results from the experiment is to determine the most influential variables. Determine the most influential variable using the Yates Algorithm method.
Table 5. Yates Algorithm

| Freezing Rate (g/jam) | 1 | 2 | 3 | divider | explanation                 |
|-----------------------|---|---|---|---------|----------------------------|
| 6                     | 6 | 78| 144| 8       | 18                         |
| 0                     | 72| 66| -16| 4       | -4                        |
| 42                    | 0 | -18| 132| 4       | 33                        |
| 30                    | 66| 2 | -4 | 4       | -1                        |
| 0                     | -6| 66| -12| 4       | 3                        |
| 0                     | -12| 66| 20 | 4       | 5                        |
| 32                    | 0 | -6| 0 | 4       | 0                         |
| 34                    | 2 | 2 | 8 | 4       | 2                         |
|                       |   |   |   |         | Type, Temperature & Salinity |

Yates algorithms table above shows that the most influential variable is the salinity.

3) Result of Least Square Method

Table 6. Least Square

| Freezer Temperature (°C) | Salinity (%) | Freezing Rate (g/jam) |
|--------------------------|--------------|------------------------|
| T                        | S            | FR                     |
| -19                      | 20           | 6                      |
| -17                      | 20           | 0                      |
| -19                      | 10           | 42                     |
| -17                      | 10           | 30                     |
| -19                      | 20           | 0                      |
| -17                      | 20           | 0                      |
| -19                      | 10           | 32                     |
| -17                      | 10           | 34                     |
| Σ                         | 2600         | -2160                  |

Normal Equation:

\[ b_0 \sum T^2 + b_1 \sum TS = FRT \]
\[ b_0 \sum TS + b_1 \sum S^2 = FRS \]

From the least square table, the normal equation becomes:

\[ 2600 b_0 - 2160 b_1 = -2608 \]
\[ -2160 b_0 + 2000 b_1 = 1500 \]

To find the value of \( b_0 \) and \( b_1 \) use the Matrix, so that it is obtained:

\[ b_0 = -3.6976 \]
\[ b_1 = -3.24341 \]

So the equation obtained is:

\[ FR = -3.697 T - 3.243 S \]

4) Response Surface Methodology (RSM)

a) Krosok
The first step is to use the initial first order method. This step takes 7 times experiments.

Table 7. Krosok Initial First Order Results

| No | Salinity (%) | Temperature (°C) | Ice Mass (gram) | Freezing Rate (g/hour) |
|----|--------------|------------------|-----------------|------------------------|
| 1  | 20           | -19              | 30              | 6                      |
| 2  | 20           | -17              | 0               | 0                      |
| 3  | 10           | -19              | 210             | 42                     |
| 4  | 10           | -17              | 150             | 30                     |
| 5  | 15           | -18              | 80              | 16                     |
| 6  | 15           | -18              | 90              | 18                     |
| 7  | 15           | -18              | 90              | 18                     |
Determination of angles used using calculations:

\[
\Delta X = \frac{-36 + (-30)}{2} = -33
\]

\[
\Delta Y = \frac{-12 + (-6)}{2} = -9
\]

\[
\alpha = \tan \frac{\Delta Y}{\Delta X}
\]

\[
= \tan \frac{-9}{-33} = 15.255
\]

b) Salt

The step of this type of salt is same with Krosok. The first step is to use the initial first order method. This step takes 7 times experiments.
Table 8. Salt Initial First Order Result

| No | Salinity (%) | Temperature (°C) | Ice Mass (gram) | Freezing Rate (g/hour) |
|----|--------------|------------------|-----------------|------------------------|
| 1  | 20           | -19              | 0               | 0                      |
| 2  | 20           | -17              | 0               | 0                      |
| 3  | 10           | -19              | 160             | 32                     |
| 4  | 10           | -17              | 170             | 34                     |
| 5  | 15           | -18              | 100             | 20                     |
| 6  | 15           | -18              | 110             | 22                     |
| 7  | 15           | -18              | 100             | 20                     |

Determination of angles used using calculation:

\[
\Delta X = \frac{-32 + (-34)}{2} = -33
\]
\[
\Delta Y = \frac{-2 + 0}{2} = -1
\]
\[
\alpha = \tan \frac{\Delta Y}{\Delta X} = \tan \frac{-1}{-33} = 1.74
\]
5. Conclusions

In both types of salt cannot carry out RSM experiments for the first order secondary steps. This is caused by the following [3, 4, 5]:

a. The results show that the value continues to decrease with a maximum freezing rate of 50 g/hour and constant for Krosok and 48 g/hour and continues to decrease to 50 g/hour constant for salt.

b. Change in the percentage of salinity is limited because if the water adds more salt, it will become saturated so that it cannot dissolve completely.

c. The freezer temperature changes are limited according to the specifications of the refrigerator.

On the graph of the Krosok, the freezing rate tends to the bottom left at an angle of 15.25°. On the graph of the salt, the freezing rate tends to the upper left with an angle of 1.74°. This indicates that the salinity is more influential than the temperature of the freezer.

The results obtained for both types of salt are, the lower the salt content, the faster the freezing rate. As for the type of salt, the fastest freezing rate according to the results of the data obtained is salt.

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