Experimental Design Lab #1

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OBJECTIVES

- Introduce students to factorial design of experiments
- Use mathematical equations to determine factor significance
- Develop models using factorial design results

INTRODUCTION

Experimental design (or Design of Experiments, DOE) is an important tool for engineers that allows for an accurate cause and effect analysis with the fewest number of experiments. There are three important definitions for experimental design:

> **Factors** are the independent variables that will be changed in order to determine their effects;
>
> **Levels** are the different values of the factors to be evaluated;
>
> **Responses** are dependent variables that are measured after changing the levels of the factors.

Choosing appropriate factors, levels, and responses is the most important part of the design because this dictates whether the results are useful or not. Preliminary testing is often required to figure out the minimum and maximum levels of the factors that result in a measureable response.

Experimental designs are used appropriately when there is no existing theory or basic principles that predict the outcome. An inappropriate experimental design would be creating a mixture of oil and vinegar and measuring the mass % of vinegar as the response. Using the basic definition of mass %, it is simple to calculate the mass % of vinegar in the mixture without having to physically do the experiment. In addition, experimental designs should have at least two factors to make the design meaningful. An experiment with one factor is relatively straightforward and does not require a rigorous experimental design.

For example, if you wanted to see effect that the initial temperature and cooking time have on the temperature of microwaved pizza, you could create an experimental design with 2 factors at 2 levels each with 1 response. The 2 factors are initial temperature and cooking time. The two levels of initial temperature could be room temperature and the original frozen temperature. The levels of cooking time could be 3 minutes and 5 minutes. The response in this case is the final temperature of the microwaved pizza. The objective of this experiment is to find a mathematical relationship final temperature as a function of initial temperature and cooking time. This would be useful in predicting the amount of time needed to cook a pizza initially at a different temperature to a new desired final temperature, for example.

Translating this scenario into typical design of experiments representations produces the following table and plot.

Table 1: Experimental design using coded values

Initial Temp	Cooking Time
-1	-1
+1	-1
-1	+1
+1	+1

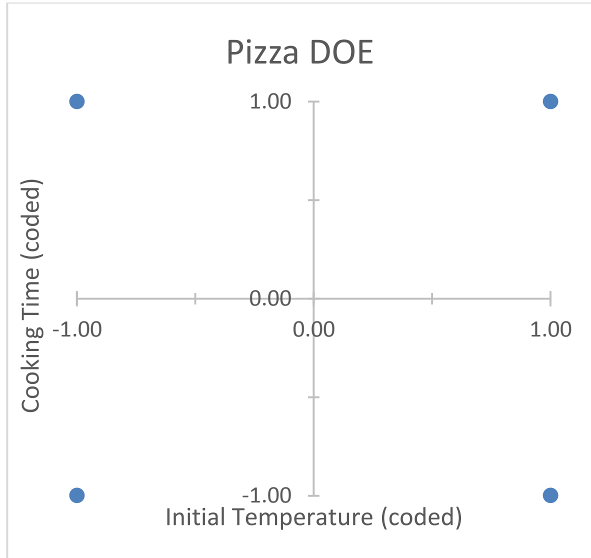


Figure 1: Graphical representation of experimental design with each dot representing a run

The -1 and +1 in Table 1 are called coded variables. For the initial temperature, the -1 represents frozen (0°C) and the +1 represents room temperature (20°C). For the cooking time, the -1 represents 3 minutes cooking time, and the +1 represents 5 minute cooking time. In this design, there are 4 possible experiments which are called runs or trials. The number of runs can be calculated by the formula $(\text{\# of levels})^{(\text{\# of factors})}$. $2^2 = 4$

After the experiments are run and the final temperature data is collected it will look like:

Table 2: Example of data collection with one response, Final Temperature

Initial Temp	Cooking Time	Final Temp (°F)
-1	-1	70
+1	-1	90
-1	+1	100
+1	+1	120

To analyze these results, a factor effect, E, is calculated for each factor and a minimum significant factor effect (MSFE) is calculated to determine if the factor has a significant effect on the response.

To calculate the factor effect, take the average value of the responses at the +1 level minus the average value of the responses at the -1 level. The larger the difference, the larger the effect of that factor. In our example, the factor effects of initial temperature, $E_{\text{Initial Temp}}$, and cooking time, $E_{\text{Cooking Time}}$, are the following:

$$E_{Initial\ Temp} = (Avg\ of\ +1\ values) - (Avg\ of\ -1\ values) = \frac{90+120}{2} - \frac{70+100}{2} = 20 \quad (1)$$

$$E_{Cooking\ Time} = (Avg\ of\ +1\ values) - (Avg\ of\ -1\ values) = \frac{100+120}{2} - \frac{70+90}{2} = 30 \quad (2)$$

The MSFE is calculated using the following equation where t is a specified value based on degrees of freedom and confidence level, s is the standard deviation of a set of repeat runs, m is the number of “+1” of a single factor, and k is the number of replicates. For this example, t(0.05, 3) = 3.182 (95% confidence level, 3 degrees of freedom) and s is given to be 1.31.

$$MSFE = t * s\left(\frac{2}{m*k}\right)^{\frac{1}{2}} = 3.182 * 1.31\left(\frac{2}{2*1}\right)^{\frac{1}{2}} = 4.168 \quad (3)$$

If the absolute value of the factor effect is greater than the MSFE, then the factor is significant at the level of significance specified. In this example, |20| > 4.168 and |30| > 4.168 so both factor effects are significant at the 95% confidence level.

The final step in data analysis is to develop a model. The model equation is the following:

$$Response = (average\ of\ all\ repsones) + \left(\frac{Factor\ A\ Effect}{2}\right) * (coded\ Factor\ A) + \left(\frac{Factor\ B\ Effect}{2}\right) * (coded\ Factor\ B) + \cdots \quad (3)$$

$$Final\ Temperature = 95 + 10 * coded\ initial\ temp + 15 * coded\ cooking\ time$$

By inserting the coded value of initial temperature (-1 to +1) and the coded value of cooking time (-1 to +1) an estimate of the final temperature can be made. For example, for a coded value of 0 initial temperature (actually 10 °C) and 0 cooking time (actually 4 min), the estimated final temperature is 95°C.

In this lab, the class will be conducting a set of experiments to determine the effects of CMC and Poloxamer content on film thickness and surface pH using a similar design of experiments.

INSTRUCTOR'S NOTE

Student t-values for other types of experiments can be seen below

Table 3. Student t values. First row is confidence interval. First column is number of degrees of freedom.

v	0.1	0.05	0.025	0.01	0.005	0.001
1	3.078	6.314	12.706	31.821	63.657	318.313
2	1.886	2.29	4.303	6.965	9.925	22.327
3	1.638	2.353	3.182	4.541	5.841	10.215
4	1.533	2.132	2.776	3.747	4.604	7.173
5	1.476	2.015	2.571	3.365	4.032	5.893
6	1.44	1.943	2.447	3.143	3.707	5.208
7	1.415	1.895	2.365	2.998	3.499	4.782
8	1.397	1.86	2.306	2.896	3.355	4.499
9	1.383	1.833	2.262	2.821	3.25	4.296
10	1.372	1.812	2.228	2.764	3.169	4.143
11	1.363	1.796	2.201	2.718	3.106	4.024
12	1.356	1.782	2.179	2.681	3.055	3.929
13	1.35	1.771	2.16	2.65	3.012	3.852
14	1.345	1.761	2.145	2.624	2.977	3.787
15	1.341	1.753	2.131	2.602	2.947	3.733
16	1.337	1.756	2.12	2.583	2.921	3.686
17	1.333	1.74	2.11	2.567	2.898	3.646
18	1.33	1.734	2.101	2.552	2.878	3.61
19	1.328	1.729	2.093	2.539	2.861	3.579
20	1.325	1.725	2.086	2.528	2.845	3.552

MATERIALS NEEDED

- 1000 mL beaker
- Hot plate and mixer
- Magnetic stir bar
- CMC (carboxymethyl cellulose)
- Poloxamer 188
- Sodium lauryl sulfate
- Citric acid (anhydrous)
- Glycerol

- Sucrose
- Peppermint oil
- Dropper
- Deionized water
- 3 mL syringe
- 2 Büchner (vacuum) flasks
- Vegetable oil cooking spray
- Funnel
- Fine mesh screen
- Vacuum tubing
- Vacuum source
- Spatula
- Stainless steel apparatus
- Tubing and stoppers
- Blue food dye (Blue #40

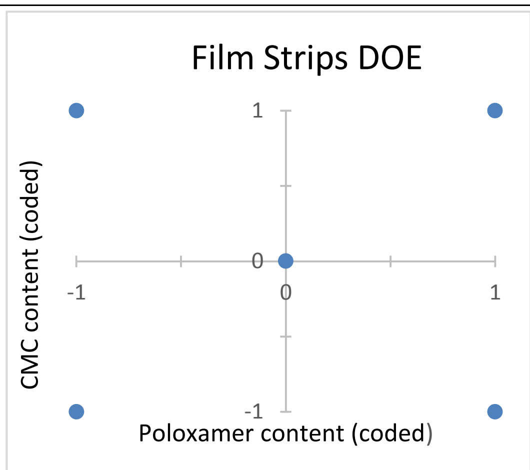
SAFETY CONDITIONS

Wear safety glasses at all times while within the designated lab area. Wear gloves if necessary.

PROCEDURE

Experimental Design

The factorial design for this experiment is provided below:



Trial	CMC	Polox.
1	-1	-1
2	1	-1
3	-1	1
4	1	1

Figure 2: Graphical representation of film strip experimental design (left) and coded values of carboxymethyl cellulose and poloxamer 188 (right)

Table 3: The coded values of the polymers correspond with actual amount of polymer in grams

CMC coded	**CMC actual (g)**	**Polox coded**	**Polox actual (g)**
1	3	1	3
-1	2	-1	2
0	1.5	0	1.5

Film Creation

Species	Weight (g)	Weight %
CMC	DOE	DOE
Poloxamer 188	DOE	DOE
Glycerol	2.4	0.6
Peppermint oil	0.4	0.1
Citric acid	0.8	0.2
Sodium lauryl sulfate	0.8	0.2
Sucrose	1.2	0.3
Water	400	97

Groups and Trial Numbers

Group Number	Week 1 Trial	Week 2 Trial
1	1	3
2	2	4
3	3	1
4	4	2
5	1	3
6	2	4

1. Weigh out the appropriate amounts of all powdered ingredients.
2. Add the required amount of deionized water to the large beaker. Reminder: density of water = 1 g/mL.
3. Place the beaker on the hot plate and add the stir bar. Set the heat to the lowest setting and set the stir to a low-medium rate (4 out of 10).
4. Add the CMC and Poloxamer to the water at a very slow rate, dusting the powder over the surface of the water and waiting for it to be absorbed. Once most of it is

mixed in, the solution will become very viscous and trap air bubbles. Once the viscosity increases, you will need to increase the stirring intensity. Do this slowly.

5. Add the glycerol to the solution with the 3 mL syringe. You will need approximately 2 mL of glycerol to correspond to 2.4 grams of glycerol.
6. Add the remaining components to the solution similarly to how the polymers were added. At this point, the solution should be viscous and appear opaque white.
7. Add three drops of peppermint oil to the solution.
8. Add one drop of blue food dye. The mixture should now be a light blue color.
9. Transfer the solution into the vacuum flask with the mesh and funnel, pouring through the mesh, to catch any large clumps of solidified product and the stir bar. Discard the solidified product.
10. We will now make a vacuum filtration system. The purpose of this is to de-aerate the mixture. This minimizes the bubbles in the solution. Hook the vacuum flask up to a tube and place a rubber stopper in the top of the flask. Then, connect the tube to the other vacuum flask. Next, place a stopper with an attachment into the top of the other flask and connect this to the vacuum source. This second beaker will stop any foam from entering the vacuum.
11. Turn on the vacuum and wait approximately 30 minutes for the gas to leave the solution. The solution should slowly turn clear and may get frothy. The froth will subside.
12. Turn off the vacuum and disconnect the tubing from the vacuum source. Then, remove the beaker with solution from the setup.
13. Spray a paper towel with vegetable oil cooking spray and wipe container with paper towel. This helps to reduce the chance of the solution sticking to the container.
14. Evenly pour out the solution into the container
15. Allow 1-2 days for the samples to dry. The batch should appear much thinner and have a glossy finish on its surface.

Film Thickness

1. Carefully cut out four 1 inch x 1 inch square of film from each quadrant of the film sheet
2. Using a caliper, take each sample and place it in the jaws of the caliper.
3. Adjust the jaws so that the sample fits snugly between them. Do not over tighten the caliper so that the sample tears. The sample should be pinched, but also be able to slide out from between the jaws when a small force is applied to it.
4. Record your results and repeat for all samples.

Surface pH

1. Using one of the halves from each sample, use a pipette to drop a small quantity of DI water on the strip.
2. Place a broad-range litmus paper strip in the drop.
3. Compare the color of the strip to the package to determine the pH of the sample.
4. Record your results and repeat for the rest of the samples. Again, you only need to measure the pH from one half of each sample.

DATA ANALYSIS

Using the gathered data from all trials and Equations 1 and 2 shown in the Introduction section, calculate the effect of CMC on the thickness, $E_{CMC,Thickness}$, the effect of Poloxamer on thickness, $E_{Polox,Thickness}$, the effect of CMC on the surface pH, $E_{CMC,pH}$, and the effect of Poloxamer on the surface pH, $E_{Polox,pH}$.

Next calculate the minimum significant factor effect (MSFE) using Equation 3. Use t = 3.182. Calculate s for thickness and pH by using the set of data collected from the center point. Compare the absolute value of the effect, E, to the MSFE to see if any factors have a significant effect on the responses pH and thickness.

CMC	**Polox**	**Folding**	**pH**	**Thickness (in)**			
1	**-1**	**11.0**	**4.4**	**0.0046**	**0.0045**	**0.0051**	**0.0058**
1	**1**	**1**	**4.2**	**0.015**	**0.014**	**0.021**	**0.0115**
-1	**1**	**10**	**4**	**0.016**	**0.015**	**0.0155**	**0.017**
-1	**-1**	**6**	**4**	**0.009**	**0.009**	**0.007**	**0.008**
0	**0**	**11**	**4.4**	**0.008**	**0.009**	**0.007**	**0.008**

Develop a model using Equation 4 for the factors that have a significant effect on the response.

QUESTIONS

1. Which factor effects are significant for thickness? For surface pH? Explain why you think the factor effects are significant or not. Do your results make sense?

pH		
S+	**8.6**	**8.2**
S-	**8**	**8.4**
E	**0.316667**	**-0.11667**
MSFE	**0.434744**	
Sig?	**No**	**No**
s	**0.136626**	
Thickness		
S+	**0.0815**	**0.125**
S-	**0.0965**	**0.053**
E	**0.057375**	**0.018**
MSFE	**0.000849**	
Sig?	**Yes**	**Yes**
s	**0.000534**	

The amount of polymer does not have a significant effect on surface pH. This makes sense because there is the same amount of citric acid in all samples. The amount of polymer does have a significant effect on film thickness. This also makes sense because the more polymer added, the more water that is retained in the film.

2. a) The center point (0,0 in coded variables) was measured to have a pH of 4.4 and thickness measurements of 0.008 in, 0.009 in, 0.007 in, and 0.008 in. Use these center point results to test the significant models. What is the percent error between the model value and actual value? Is this acceptable?

$$\boldsymbol{Thickness = 0.0105\ in + 0.0287\ in * CMC + 0.009\ in * Polox}$$

$$\boldsymbol{Thickness = 0.0105\ in + 0.0287 * 0 + 0.009 * 0 = 0.0105\ in}$$

$$\%\boldsymbol{error} = \frac{\boldsymbol{0.0105\ in - 0.008\ in}}{\boldsymbol{0.0105\ in}} * \boldsymbol{100}\% = \boldsymbol{23.8}\%$$

b) Sometimes factors do not have a linear effect on the response. Test this in this case by calculating the minimum significant curvature effect (MSCE) using the following equation:

$$MSCE = t * s * \sqrt{\frac{1}{m * k} + \frac{1}{c}}$$

Where *t, s, m, k* are the same variables as in Equation 3 and *c* is number of center points. Compare the MSCE to the curvature effect which is calculated the following equation:

$$Curvature\ Effect = Average\ Corner\ points - Average\ Center\ points$$

$$\boldsymbol{MSCE = 3.182 * 0.000534 * \sqrt{\frac{1}{2 * 4} + \frac{1}{1}} = 0.001802}$$

$$\boldsymbol{Curvature\ Effect = \ 0.011125 - 0.008 = 0.00312}$$

The model has significant curvature.

3. Umbrella Corp. decides to start making a long-lasting dissolvable strip with an anti-viral drug to help treat a spreading virus. It was found that the thickness of the film and the time it takes to dissolve can be modeled as a linear relationship:

$$D = -4 + 1400 * T$$

Where T is the thickness of the strip (inches) and D is the duration time (min) of the strip. The films are made with equal amounts of CMC and Poloxamer 188. The company wants to develop a strip that lasts 20 minutes. Recommend a formulation to make a batch of 10 kg of film solution.

$$\boldsymbol{20\,\mathrm{min} = \ -4\frac{min}{in} + 1400 * T}$$
$$\boldsymbol{T = 0.016\ in}$$
$$\boldsymbol{0.016\ in = 0.0105\ in + 0.0287\ in * CMC + 0.009\ in * Polox}$$
$$\boldsymbol{CMC_{coded} = Polox_{coded} = 0.146 = 2.57g}$$

Species	Weight (g)
CMC	2.57
Poloxamer 188	2.57
Glycerol	60
Peppermint oil	10
Citric acid	20
Sodium lauryl sulfate	20
Sucrose	30
Water	9700