Design of Experiments Lab #2
Developed by: Mike Evangelista, Nathan Haden, Alex Jannini, Rowan University, Department of Chemical Engineering
Edited by: C. Stewart Slater and Mariano Savelski, Rowan University, Department of Chemical Engineering
Date of Experiment:

OBJECTIVES

- Reinforce design of experiments theory
- Make individual factorial designs and conduct analysis on responses

INTRODUCTION

Creating experimental designs is often the most important part of a project. A good experimental design should focus on proving the validity of an existing hypothesis. This means that the experimenter should already have an educated guess as to what effect changing a factor will have on a specific response. For more background information on the Design of Experiment (DOE), refer to the introduction section in DOE Laboratory #1.

For this laboratory, you will design your own 2^2 factorial using the thin dissolvable strips to test a specific response and a function of several different factors.

You will be picking **two factors** and **one response** with another team. There should be a valid hypothesis as to why the specific factors and corresponding response are chosen. You will then need to design a factorial experimental design with two factors at two levels. Refer to Table 1 for suggested levels of the factors. Your team, and the team you are collaborating with, should discuss how to split up experimentation. This will need to be approved by the instructor before you can begin experimentation.

Table 1. Factors at suggested limits and responses

Factors	Limits	Responses
Water	400 g ± 100 g	Thickness
CMC	2.5 g ± 0.5 g	Moisture Content
Poloxamer 188	2.5 g ± 0.5 g	Folding Endurance
Sodium Lauryl Sulfate	0.82 g ± 0.27 g	Surface pH
Glycerin	2.4 g ± 0.8 g	Absorbency Test
Sucrose	1.27 g ± 0.42 g	
Citric Acid	0.82 g ± 0.27 g	

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)
- Spectrophotometer
- pH testing paper

SAFETY CONDITIONS

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

PROCEDURE

Film Creation

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/or 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. Reminder: the density of glycerol at room temperature is about 1.26 g/mL.

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. NOTE: If you are conducting a degradation analysis, you will need to use Blue Food Dye #1, with the spectrophotometer set to 630 nm.
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. If your group's chosen response is moisture content, skip to the Moisture Content section, otherwise continue to step 14.
14. 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.
15. Evenly pour out the solution into the container
16. Allow 1-2 days for the samples to dry. The batch should appear much thinner and have a glossy finish on its surface.

Moisture Content

1. Carefully pour some of the solution into a 500 mL graduated cylinder. This will make it easier to transfer the solution to the container or petri dish.
2. Take a petri dish and weigh it. Record this weight.
3. Prepare a sample of the film in the petri dish by adding 10 mL to the dish. Do this by using a small graduated cylinder (10 to 25 mL). If any bubbles remain on top of the solution, be sure to draw solution from under the surface.
4. Weigh the wet petri dish and record this weight.
5. Allow 1-2 days for the samples to dry. Weigh the petri dish again and record the difference in weight.

Folding Endurance

1. Carefully cut out six 1 inch x 1 ½ inch squares of film

2. While wearing gloves, fold the film in half and firmly crease along the fold.
3. Unfold the film and refold it the other way, along the same fold. Firmly crease.
4. Repeat steps 2 and 3 until the film breaks and record the amount of folds. Stop if the number of folds exceeds 50.

Film Thickness

1. Carefully cut out six 1 inch x 1 ½ 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.

Absorbency Test

1. Start by turning on the spectrophotometer and setting it to take absorbance readings at 630 nm.
2. While the spectrophotometer warms up, place 2.5 mL of deionized water into each of the petri dishes.
3. Place one of the petri dishes into the oven/incubator that is set at 37°C.
4. Allow 20 minutes to pass so that the water may reach 37°C and the spectrophotometer may warm up.
5. After the 20 minutes have passed, fill a cuvette with deionized water and zero the spectrophotometer.
6. Now that the spectrophotometer has been zeroed, use the tweezers to place a strip into one of the petri dishes. Once the strip has been placed in the petri dish, cover and start the timer.
7. Place the other strip in the other petri dish using the tweezers. Once placed, close the petri dish and start a secondary timer. NOTE: It is a good idea to stagger the starting times so that you do not find yourself rushing to take two measurements.

8. After five minutes have elapsed, take a sample of your water into a cuvette. Make sure that this sample is relatively far from the dissolvable strip so that you do not accidentally pick up any large portions of the dissolvable strip.
9. Take an absorbance reading then return the sample to the corresponding petri dish.
10. Take absorbance readings every five minutes for the first thirty minutes. After that, take absorbance readings every ten minutes until you have reached 90 minutes. Record your results.

DATA ANALYSIS

Calculate the factor effect, *E*, for each factor using Equation 1.

$$E_{Factor} = (Avg\ of\ +1\ values) - (Avg\ of\ -1\ values) \quad (1)$$

Compare the absolute value of *E* to the Minimum Significant Factor Effect (*MSFE*) which can be calculated using Equation 2. The t value is based on the degrees of freedom and confidence level. For this laboratory t = 3.182. The *s* value is the standard deviation of a single data point. Calculate the standard deviation, *s*, using the response values of the center point in your design.

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

If the absolute value of the factor effect is greater than the MSFE, then the factor is significant. The final step in data analysis is to develop a model. The model equation is the following:

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

FINAL PROJECT

A presentation summarizing the experimental design, factors, responses, and results will be made and presented to the class as a final project. The presentation should address the following questions:

What factors and responses were chosen? What was the reasoning behind the choices? Is there any existing theory model that can help predict the effect of the factor on the response?

Show the data that was collected. Does it makes sense? Is there any outliers or trends in the data that need to be addresses before analysis?

Which factors had a significant effect on the response? Was this result expected? Explain why you think the factor is significant.

Create the response model as a function of the coded factor effect. What could be done to improve the model?

What conclusions can be made of the experimental design?

Include any pictures, tables, or figures that are relevant to explain the experimental design.