Names of Students:	Instructor:
Course:	Date:

#### Introduction

Nature has demonstrated evidence for self-healing in biological systems. In fact the human body has the ability to undergo self-healing through a series of chemical reactions. Industry is now looking to capitalize on the complex physiology that biology has been able to demonstrate in nature. Imagine material that have the ability to repair themselves. Although their is some technology that currently exists possessing the ability to undergo a self healing process scientist and engineers are working on improving the previous methods and devising new more efficient methods. During this laboratory experience students will use basic concepts from their chemistry course to engineer and test a new type of polymer that has the ability to repair itself when severed.

Encapsulated polymers have many applications from drug delivery to self-healing materials. Capsules can be designed in a variety of different ways from encapsulating the polymer to using the polymer to form the capsule around a compound. There is a research group out of The University of Akron working on encapsulating an Alkyd polymer inside of a silica capsule to be used as an additive in coating that will give the coating self-healing properties. In this lesson students will investigate the encapsulation process by using a polymer sodium alginate to form a capsule around fruit juice by crosslinking the polymer.

The two polymers the students will be working with are gelatin and sodium alginate. The students will use concepts from the lecture to design alginate fruit juice spheres through a direct food spherification by submerging the sodium alginate solution into a calcium lactate bath allowing for chemical crosslinking to occur on the outside of the alginate juice. Once the spheres are made the students will then prepare the gelatin solution making sure to prepare one control sample along with the experimental samples by adding calcium lactate solution to the gelatin mix to ensure that calcium is present in the gelatin mold. The students can then add their alginate juice spheres to the gelatin mold.

Gelatin strength will be evaluated by using a durometer to perform a Bloom Test for hardness and self healing will be checked by slicing the gelatin with a knife. Bloom Test will be used to check the hardness of the self healed gelatin. If the gelatin splits during the second Bloom Test the gelatin will be considered to fail and the force should be recorded at which the gelatin failed. The durometer will be used to measure the rupture force of the gelatin and then again once the gelatin has healed. At this point the students will report their data on the laboratory worksheet and answer the questions.

### Pre Laboratory Questions (Answer the following Questions):

- 1) What is the chemical formula for sodium alginate?
- 2) Draw the chemical structure of sodium alginate?
- 3) What is the molar mass of sodium alginate?
- 4) What is the chemical structure of calcium lactate?
- 5) Draw the chemical structure of calcium lactate?

- 6) What is the molar mass of calcium lactate?
- 7) Write an Equation to convert from mass to weight?
- 8) How does Bloom number relate to average molecular mass?

9) How much water would need to be added to 2 g of calcium lactate (assume the calcium lactate occupies 2 ml) to make a 0.046 M solution of calcium lactate and water? *Research may be needed for this.*

- 10) What is the group's strategy to design the self healing gelatin?
- 11) What variations will the group make between the molds?

12) What is the control, independent, and dependent variables for this laboratory?

13) What is the groups hypothesis for this laboratory?

### **Materials and Methods**

#### For Synthesizing Self Healing Gelatin

- Sodium Alginate
- Calcium Lactate
- Gelatin
- Blender
- 12 Cup Pan / Gelatin Shot Cups
- 500 ml beakers
- 10 ml Food Syringes with Attachments
- Mesh Strainer
- 500 ml Measuring Cups

#### Synthesis Procedure

#### <u>Day 1</u>

Spherification synthesis

• Plastic Spoons / Knives

### Equipment for Testing

- Scalpel
- Modified gel property testing device (scale with a Clamp attached)
- Cooking spatula
- Precise weighing scale

- Prepare a 0.46 M solution of calcium lactate and Water (if your group decides to vary the concentration please specify in your lab report).
  Remember M= mols of solute/ L of Solution
- 2) Create the alginate juice spheres by using the tools available, select the pre-made 0.025 M alginate juice solution and drip it into a 200 ml 0.046 M calcium lactate bath (students can vary the time they leave the spheres in the bath recommended 2 minutes- please record the time the spheres were left in the bath). Their is the ability to make 11 different types of spheres (keep in mind that concentration of spheres might want to be studied along with shape).
- 3) Recover the spheres from the calcium lactate bath using the mesh strainer. Rinse the spheres in a water bath (leave in the bath for a minimum of 30 seconds).
- 4) Using graph paper/ruler measure the height and diameter of the spheres. Record the data for the spheres in the correct sample location.
- 5) Make sure that a few extra are made and put to the side to be tested later today. Make sure to measure diameter.

#### Gelatin Synthesis

1) Make the gelatin mix using the bunsen burner/hot plate to boil the water. Following the directions on the package for the amounts of boiling water and gelatin mix (record your quantities in the lab report). Do not add the cold water yet.

- 2) Decide if the group will use a mold tray or individual gelatin shot cups. Label each cup/mold, so that it can be identified what experimental quantities are in each.
  - I. In one of the molds prepare a control sample of gelatin based off of the recommended ratio of warm water-gelatin mix to cold water (no calcium lactate added).
  - II. Prepare 0.025 M calcium lactate solution with 200 ml of cold water. Notice new calculations will need to be performed.
  - III. In the other molds add and vary the concentrations of warm water-gelatin mix to cold calcium lactate solution (record the concentrations). Make sure to stir the solutions.
  - IV. Add the spheres to the designated experimental molds ensuring that the quantity is recorded. Their is the ability to experiment with 11 samples and still have one control. **Be sure to save a few of the polymer spheres to test.**
- 3) Place molds into the refrigerator at  $10^{\circ}$ C.
- 4) Record any observations.
- 5) Clean up and put the equipment back.

#### Testing the Capsule Strength

- 1) Take a capsule (polymer sphere) and place it on the durometer device.
- 2) Adjust the clamp to just before it makes contact with the capsule.
- 3) Zero out (tare) the scale.
- 4) Tighten the clamp and record the value measured by the scale after every turn until the capsule breaks. Make sure the value is recorded that was observed when the capsule failed.
- 5) Clean the instrument after every use.
- 6) Then convert these values to forces by multiplying them by acceleration due to gravity.
- 7) Graph the data.

### <u>Day 2</u>

#### Testing the Capsule Strength

- 1) Take a capsule and place it on the durometer device.
- 2) Adjust the clamp to just before it makes contact with the capsule.
- 3) Zero out (tare) the scale.
- 4) Tighten the clamp and record the value measured by the scale after every turn until the capsule breaks. Make sure the value is recorded that was observed when the capsule failed.
- 5) Then convert these values to forces by multiplying them by acceleration due to gravity.

#### Bloom Test of the Control and Modified Gelatin Before the Capsule is Broken

- 1) Take out the Gelatin from the mold and place it on the durometer.
- 2) Adjust the clamp to just before it makes contact with the capsule.
- 3) Zero out (tare) the scale.
- 4) Tighten the clamp and record the value measured by the scale every 1 mm until the gelatin has been displaced 4 mm and reverse the process back until it has returned to its normal state.
- 5) Record the data.
- 6) Repeat this procedure for every sample.
- 7) Make sure to test the control.

### Demonstrating the Self-Healing Gelatin

- 1) Take the Gelatin molds and a scalpel slice the gelatin mold so that it breaks open the embedded capsule.
- 2) Allow the fluid to leak out and interact with the gelatin.
- 3) Make sure that the two sides of the sliced gelatin are in contact with each other and allow two minutes for them to interact before touching it.
- 4) Using a cooking spatula place the mold back on the durometer.
- 5) Repeat the bloom test record the value measured by the scale every 1 mm until the gelatin has ruptured.
- 6) If the modified gelatin stays together for the 4 mm depression record the Bloom number, and continue to turn the clamp recording the value after every turn until the sample ruptures again (make sure to convert from mass to weight).
- 7) Repeat for every sample possible.
- 8) Make sure to test the control.

### Additional Test: Use the Durometer to Test the Rupture Force of the Modified Gelatin

- 1) If there is an intact region of the gelatin mold after self healing has been tested place that region back in the durometer.
- 2) Adjust the clamp to just before it makes contact with the capsule. Make sure that the clamp is positioned over the repaired gelatin.
- 3) Zero out (tare) the scale.
- 4) Repeat the bloom test record the value measured by the scale every 1 mm until the gelatin has ruptured.
- 5) Record the data (make sure to convert from mass to weight).
- 6) Repeat for every sample possible.
- 7) Make sure to test the control.

# Results

# Synthesis Procedure

Mass In grams of Calcium Lactate	g
Volume of Water	L
Concentration of Calcium Lactate Solution	М

# Capsule Strength

Sphere Sample	Shape	[SA]	Type of Juice	Mass (g)	Height of Spheres (mm)	Diameter of Spheres (mm)	Rupture Force (N)
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Draw a Picture of the Sphere Shapes used:

### <u>Gelatin Test</u>

Sample	[SA]	Type of Juice	[CL]	[G] & Volume	[CL] Added to G & Volume	Sphere Sample	Mass of Added Spheres Combined	Number of Spheres Added	Bloom Number Before SHT (N)	Bloom Number After SHT (N)	Rupture Force After SHT (N)	Rupture Force of Gelatin (N)
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SA = Sodium Alginate

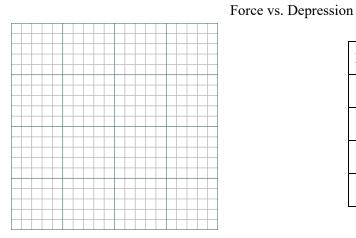
CL = Calcium Lactate

G = Gelatin (warm water plus Gelatin mix)

SHT = Self Healing Test

Draw a picture of the mold setup:

Graph & Table: Bloom Test of the Control Before Self Healing Test (Record Qualitative Observations to the Right of the Table & Include a Drawing of The Mold)



Force (N)	Depression (mm)
	1
	2
	3
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Graph & Table: Bloom Test of the Control After Self Healing Test (Record Qualitative Observations to the Right of the Table & Include a Drawing of The Mold

Force vs. Depression

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Force (N)	Depression (mm)

# Graph & Table: Rupture Test of Control (Record Qualitative Observations to the Right of the Table & Include a Drawing of The Mold

Force vs. Depression

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Force (N)	Depression (mm)	Force (N)	Depression (mm)

Graphs and Tables: Bloom Test of the Modified Gelatin Before Self Healing (Record Qualitative Observations to the Right of the Table & Include a Drawing of The Mold

Sample 2: Force vs. Depression

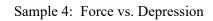
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Sample 3: Force vs. Depression

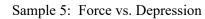
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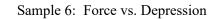
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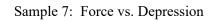
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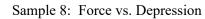
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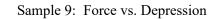
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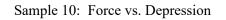
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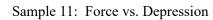
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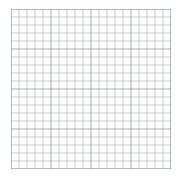
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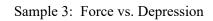
Graphs and Tables: Bloom Test of the Modified Gelatin After Self Healing (Record Qualitative Observations to the Right of the Table & Include a

### Drawing of The Mold

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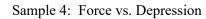
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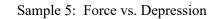
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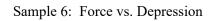
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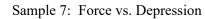
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Force (N)	Depression (mm)



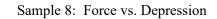
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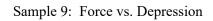
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Force (N)	Depression (mm)



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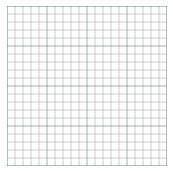
Force (N)	Depression (mm)



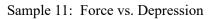
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Force (N)	Depression (mm)

Sample 10: Force vs. Depression



Force (N)	Depression (mm)



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Force (N)	Depression (mm)

Graphs and Tables: Rupture Force Measurements of the Modified Gelatin After Self Healing (Record Qualitative Observations to the Right of the Table & Include a Drawing of The Mold

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Sample 2: Force vs. Depression

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	Force (N)	Depression (mm)	Force (N)	Depression (mm)

Sample 3: Force vs. Depression

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Force (N)	Depression (mm)	Force (N)	Depression (mm)

#### Sample 4: Force vs. Depression

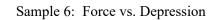
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Force (N)	Depression (mm)	Force (N)	Depression (mm)

# Sample 5: Force vs. Depression

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Force (N)	Depression (mm)	Force (N)	Depression (mm)

Sample 7: Force vs. Depression

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Force (N)	Depression (mm)	Force (N)	Depression (mm)

Sample 8: Force vs. Depression

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Force (N)	Depression (mm)	Force (N)	Depression (mm)

Sample 9: Force vs. Depression

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Force (N)	Depression (mm)	Force (N)	Depression (mm)

Sample 10: Force vs. Depression

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Force (N)	Depression (mm)	Force (N)	Depression (mm)

Sample 11: Force vs. Depression

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Force (N)	Depression (mm)	Force (N)	Depression (mm)

Graphs and Tables: Rupture Force Measurements of the Modified Gelatin Before Self Healing (Record Qualitative Observations to the Right of the Table & Include a Drawing of The Mold

Sample 2: Force vs. Depression

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Force (N)	Depression (mm)	Force (N)	Depression (mm)

Sample 3: Force vs. Depression

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Force (N)	Depression (mm)	Force (N)	Depression (mm)

Sample 4: Force vs. Depression

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Force (N)	Depression (mm)	Force (N)	Depression (mm)

Sample 5: Force vs. Depression

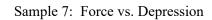
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Force (N)	Depression (mm)	Force (N)	Depression (mm)

Sample 6: Force vs. Depression

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Force (N)	Depression (mm)	Force (N)	Depression (mm)



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Force (N)	Depression (mm)	Force (N)	Depression (mm)

Sample 8: Force vs. Depression

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Force (N)	Depression (mm)	Force (N)	Depression (mm)

Sample 9: Force vs. Depression

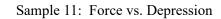
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Force (N)	Depression (mm)	Force (N)	Depression (mm)

# Sample 10: Force vs. Depression

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Force (N)	Depression (mm)	Force (N)	Depression (mm)



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Force (N)	Depression (mm)	Force (N)	Depression (mm)

Draw a picture of the capsule/capsules:

Draw a picture of the capsule/capsules embedded into the gelatin mold:

### Discussion

Answer the following questions:

1) Describe what is happening chemical to the sodium alginate juice when it is added to the calcium lactate bath. Be sure to mention cross linking (ionic/covalent and how) and talk about what is happening with the sodium and the calcium ions (how does this happen).

2) Why is it important to wash the alginate spheres in a water bath after they have been made (mention diffusion)?

3) What significance does adding the calcium lactate solution to the gelatin have in the self healing process?

4) Would the gelatin be able to show properties of self healing if it didn't have calcium lactate added to the gelatin?

5) Describe what is happening and what type of chemistry is responsible for the observed self healing in the gelatin?

6) Describe what design/combination (sphere type and gelatin/calcium lactate concentration) provided the best results?

7) What changes would be made to the design if the experiment was repeated?

8) Discuss any errors that occurred during the experiment and how they may have impacted the results?

9) Discuss whether or not the experiment was a success.

10) Moving forward how could this concept be used to engineer a new technology and provide one novel (new) concept?

### Conclusion

Write a full conclusion to this laboratory summarizing what was done, what was discovered, and what would be the next step after this to continue the work. This conclusion needs to be one paragraph (four to six sentences).