Penn State RET in Interdisciplinary Materials

Lesson/Project Plan - Teacher's Preparatory Guide

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Engineering Polymers, Synthesis and Characterization

Purpose

In this lab students will learn to engineer polymers with varying physicochemical and structural properties, synthesize using commonly available diacids and diols from bio-renewable sources via direct esterification polymerization, test their tensile mechanical properties, analyze the output data of specimens, and propose methods for improvements and future exploration.

Objectives

Students will plan and investigate the effectiveness of polymer design through mechanical testing. They will analyze, interpret, and communicate their results through the creation of a poster, delivery of scientific findings through poster presentation open to audience, and suggest future methods for exploration.

Students will also demonstrate the knowledge gained from this investigation, of how the structural integrities of monomers, can affect the properties at the bulk scale, to infer the strength of bond forces between the different monomers in a formed polymer chain.

Time required

The instructor will need one class period (45min) to introduce the project and provide critical background information. Students will need one class period (45min) to research chemicals to use, demonstrate their understanding of esterification and polymerization, and propose their resin synthesis method. Students will need 2 lab (135min) periods, to weigh out the prepolymer formulation and organize the reaction equipment setup, then to synthesis and to do the final dilution with the reactive diluent to form a UV curable resin, and another class period (45min) to create the dog bones that they will test in yet another class period (45min). Students will then be given a final lab period (90min) to analyze their results and create a final project poster to communicate their scientific findings.

Level

High school (10-12).

NGSS [9-12]

HS-PS1-3 Plan and investigate to gather evidence of how the structural integrities of monomers can affect the properties at the bulk scale, to infer the strength of bond forces between the different monomers in a formed polymer chain.

This lesson incorporates the following 3D Crosscutting Concepts:

- Patterns

- Cause and Effect
- Systems and System Models

This lesson incorporates the following 3D Science and Engineering Practices:

- Asking questions and designing problems.
- Developing and using models
- Planning and carrying out investigations
- Analyzing and interpreting data
- Constructing explanations based on evidence and suggesting solutions
- Engaging in argument from evidence
- Obtaining, evaluating, and communicating information.

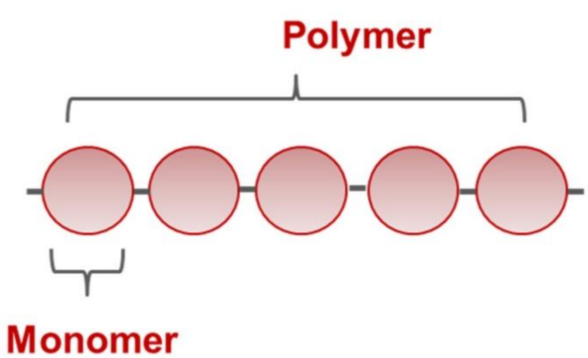
PA STEEL 3.2.9-12. B Plan and conduct an investigation to gather evidence to compare the structure of substances at the bulk scale to infer the strength of electrical forces between particles.

Teacher Background

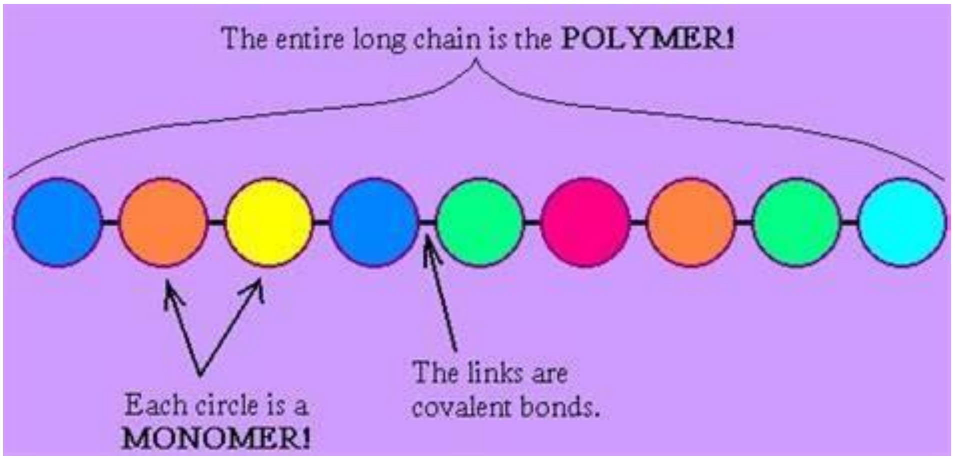
This project should be completed at the end of a high school bonding unit, so that students have in-depth knowledge of intramolecular forces, intermolecular forces, and understand the link of properties to structure for materials, including giant covalent structures. Students will apply their knowledge of bonding to this project.

The following is additional information to be communicated with students:
As the world transitions away from petrochemicals for fuel, alternatives for non-fuel petrochemical products are necessary. Many non-fuel petrochemical products contain polymeric structures - replacement of these polymers is vital to the success of the world's energy transition. The instructor should focus on the green energy transition.

Polymers are long chains of covalent molecules that have many similar subunits bonded together. These subunits are called monomers. Here is a very broad and overly general example of a polymer:

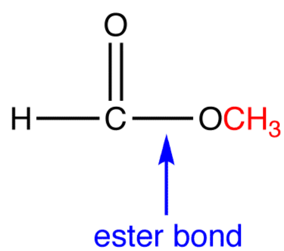


This is an example of a homopolymer, where all the individual monomers are the same. Here is another example of a polymer:



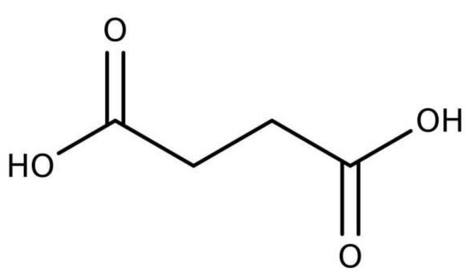
This is an example of a heteropolymer, where different monomers make up the polymer.

One common method for making polymers is through esterification. Esterification is a reaction that combines carboxylic acid group with an alcohol group forming an ester bond and water is released as a biproduct. In this example reaction, an ester bond links the monomers. Ester bonds look like:



Before we go any further, let's discuss what diacids and diols are.

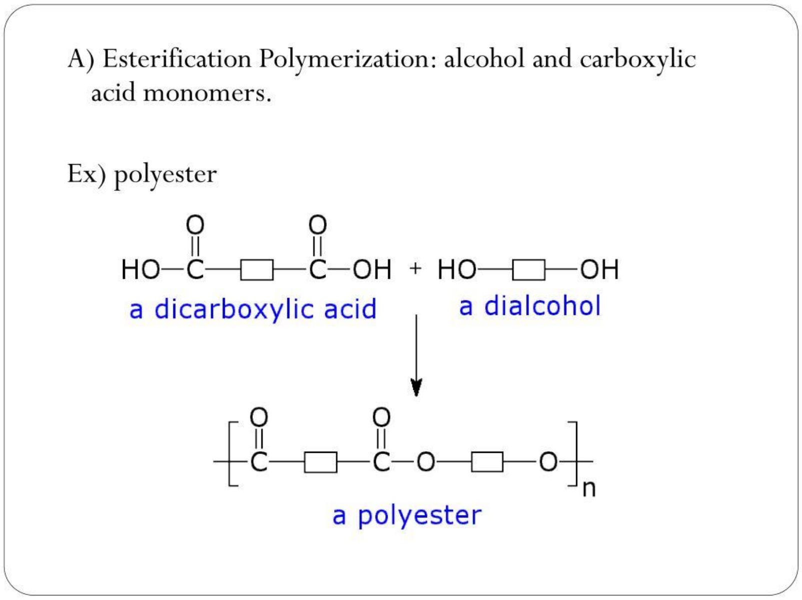
A carboxylic acid is a group containing 1 'C', 2 'O's and 1 'H' atom. An alcohol is a group of 'OH' attached to a carbon backbone. Here is an example of a dicarboxylic acid, short form is diacid (prefix di means two) that we will use in our lab. This is succinic acid:



As you can see here, a diacid contains two COOH groups on either side of the molecule.
And here is an example of a diol we will use. This is butane diol:

As you can see here, diols contain two OH groups on either side of their molecules.

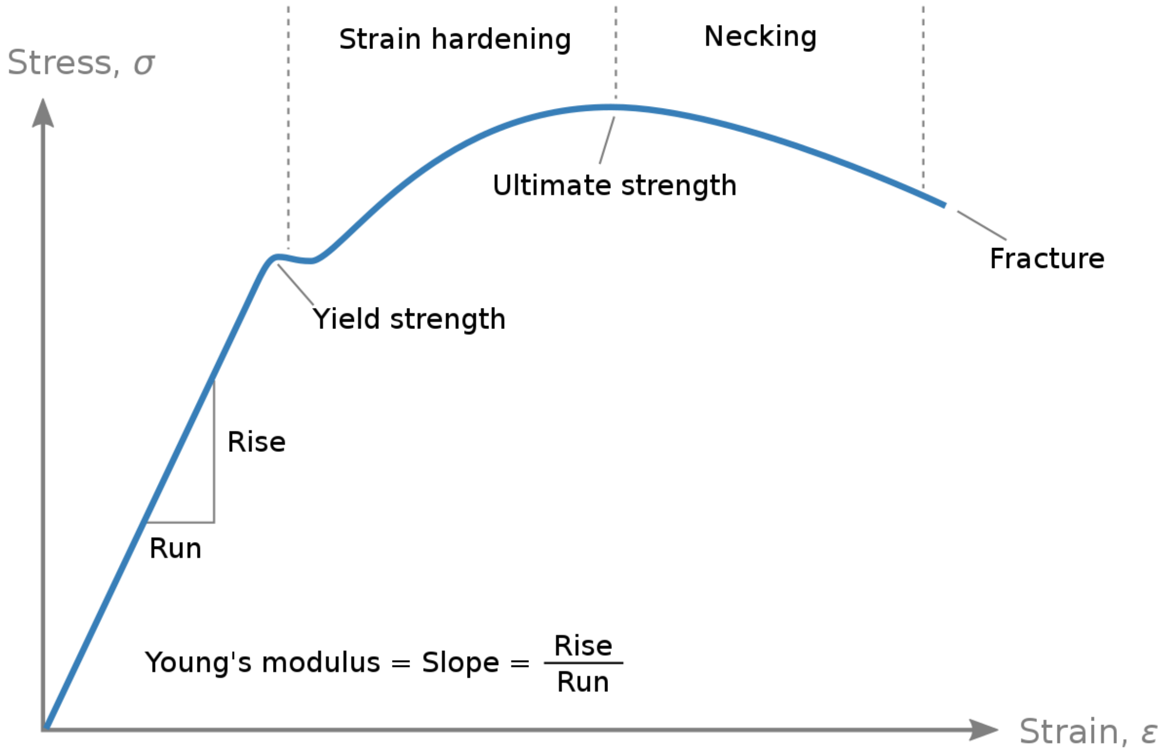
Now let's look at a reaction schematic for direct esterification:



In this reaction we take a diacid (or as named here a dicarboxylic acid) and react it together with a diol (synonym: dialcohol) to get polyester. Again, the 'ester' in polyester refers to the COO groups that exist within the polyester as a bridge between the diacid monomer and diol monomer. In this lab we will create polyesters and test their mechanical strength.

Last, we arrive at the measure of mechanical strength we will test today: Young's modulus. Young's modulus is a measure of the lengthwise tensile or compressive stiffness. It is calculated by the slope of the stress-strain curve, taken when a sample material is elongated at a constant rate. In the stress-strain curve, strain, or how much the sample deformed relative to its length, is plotted on the x axis. Strain is a unitless value, sometimes expressed as length / length or as a % of the original length of the sample. Stress is a measure of the amount of force that the material is under (in this case as it deforms) per unit area. Thus, samples must be measured at their cross section to determine stress values – force alone will not provide values for Young's modulus. An example of a stress-strain curve can be seen on the next page. In this example, the material undergoes two distinct types of elongation, elastic and inelastic. Elastic formation occurs when the sample is under low loads, where when the load is removed, the sample can return to its original shape. Elastic deformation can be seen by the first part of the curve, prior to the 'yield strength' value indicated on the graph. Young's modulus is calculated as the slope of the curve during elastic deformation of the material.

An example of such a curve can be seen here:



Similarly, inelastic deformation occurs when the material is exposed to a load that deforms the material so that it will not return to its original shape. The inelastic portion of the curve occurs in the 'strain hardening', 'necking', and fracture stages. Young's modulus is not calculated during inelastic deformation.

Materials

- Chemicals: 1,4-butanediol, 1,8-octanediol, 1,3-propanediol, itaconic acid, succinic acid, TEGDMA (Triethylene glycol dimethacrylate), and TPO
- 125 ml round-bottom flasks
- 20 ml glass vials
- Magnetic stir bars
- Transferring pipettes
- Disposable syringes
- Hot plates
- Silicone Oil
- UV lamp
- Silicone Dog bone molds
- Force sensors
- Ring stand
- Clamp
- Markers

Advance Preparation

Materials (chemicals, force sensors, hot plates, magnetic stir bars, dog bone molds, ring stands, clamps, markers etc.) can be purchased from Flinn, or Thermofischer.

Students should be expected to propose a procedure, as approved by the instructor, and acquire necessary items for that procedure when they go to do their work. More details on the requirements for this procedure can be found in the 'Lab Procedure' section below.

Safety Information

Students should ensure that they wear gloves when handling the polymers. Students should ensure they do not touch the hot plates when they are hot, and to be careful when pouring their resin into the silicone dog bone molds. All chemicals are mild skin irritants, and any skin that encounters the chemicals should be thoroughly flushed for 15 minutes with water. Proper eye wear, goggles with side shields, should be worn.

Teaching Strategies

Students should be given all the information in the teacher background section prior to the lab. This can be done through lecture format, or via note slides.

Students should work in groups of 2-3, purposefully grouped at the instructor's discretion. Students should submit a lab procedure, outlining what chemicals they want to use, and detailed procedural steps prior to starting the lab. The students' lab, in addition to the procedure, should include relevant safety information, and a blank data table that they will fill in as they work. More details on the requirements for this procedure can be found in the 'Lab Procedure' section below.

Directions for the activities

Day 1: The very first day should introduce relevant background information for the students. This can be done in lecture style, using the 'teacher background' section as a reference. Additional helpful videos on these concepts, that the teacher may want to share with students or provide these videos for remediation, include:

- Fun intro video to polymers: From DNA to Silly Putty: The diverse world of polymers - Jan Mattingly - YouTube
- Esterification reaction basics: 10 Esterification - YouTube
- The basics of Young's modulus: Understanding Young's Modulus - YouTube

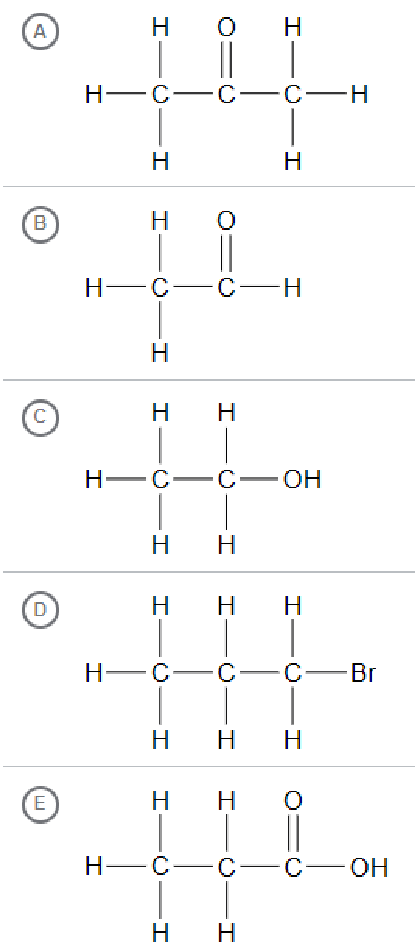
Prior to the end of the day, the instructor should issue an exit pass containing a few questions about the background material that was covered during the class period. An exit pass should include at least one question on each of the three topics covered (polymers, esterification, and Young's modulus). Remediation can be provided based on the questions that the student got incorrect, and the material that this question directly references.

A sample exit pass is provided on the next page that teachers may choose to use.

Exit Pass: Answers in red.

1. Define the term monomer in your own words: A small molecule that bonds to other monomer molecules and repeats many times to form a long polymer chain.
2. Describe the difference between a homopolymer and heteropolymer: A homopolymer is a long chain of the same monomer repeated multiple times. A heteropolymer is a polymer chain that consists of different monomers bonded together.
3. Describe how the process of esterification relates to polymerization in this lab. Be sure to clearly define what the monomers are in this reaction. An esterification reaction is the type of reaction that occurs when a carboxylic acid reacts with an alcohol to produce an ester group. In this reaction, the carboxylic acid and alcohol molecules are the monomers that will form the heteropolymer in this reaction. In this heteropolymer, it is the ester groups that link the two different monomers together.

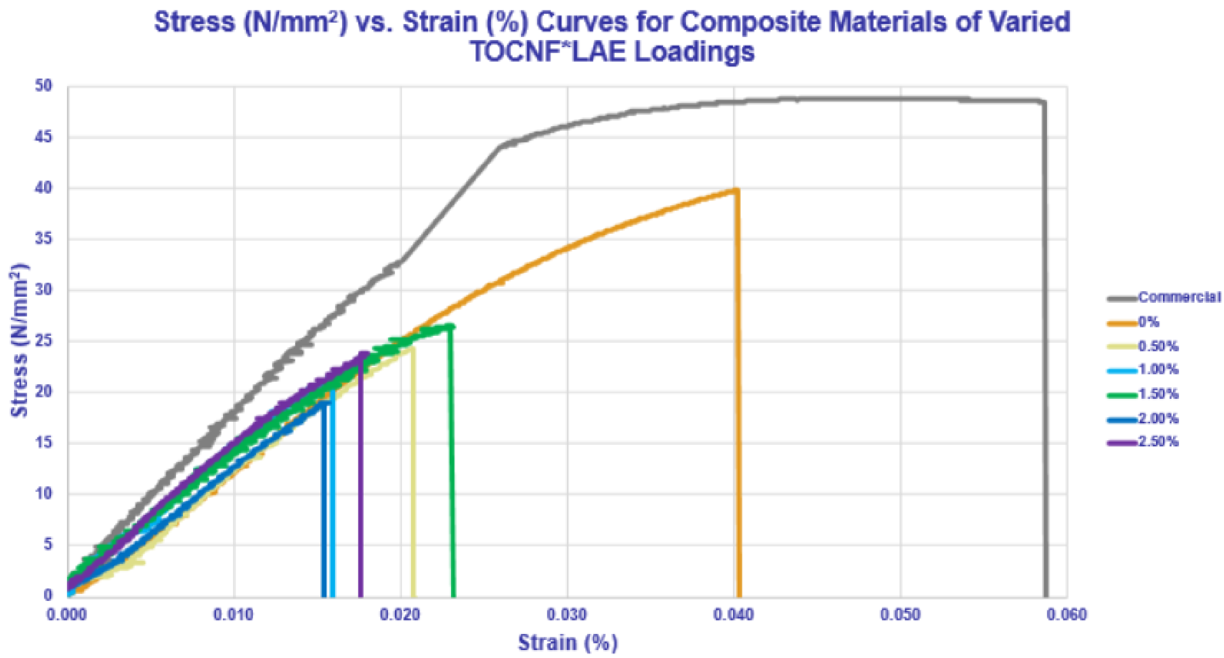
Use the following image for questions 2 through 4.



4. Which of the following molecules contain an alcohol group? Write the letter(s) of your choice(s) here. C
5. Which of the following molecules contain a carboxylic acid group? Write the letter(s) of your choice(s) here. E
6. Which of the following molecule pairs could react together to form an ester in an esterification reaction? Write the letter(s) of your choice(s) here. C & E

7. Define the terms ‘stress’ and ‘strain’. Include what units are commonly used to measure these values. Stress is a measure of the amount of force that is put upon an object per unit area. Stress is generally measured in units of Force/Area. An example is N/mm^2. Strain is a measure of the distance at which an object displaces. It is measured by dividing the amount that the object stresses divided by the length of the object and is expressed as unitless or as a percent.

The following data was taken from an experiment similar to the one you will be doing. In the graph below. Use the following graph to answer questions 8 and 9.



8. Which of the following samples has the highest value for Young’s modulus? The Commercial sample has the highest Young’s modulus.
9. Explain your previous answer, by describing how Young’s modulus can be calculated from the graph. Young’s modulus is calculated by the slope of the stress strain graph. The Commercial sample has the highest Young’s modulus on this graph as the slope of the graph is highest for the commercial sample.

Day 2: During the second day of the project, students should be given this time to do their own research into possible materials that they can choose to use for this project. By the end of this class period, students should have a working procedure that can be checked prior to the instructor as an ‘exit pass’ prior to the end of the day. Be sure to refer to the ‘lab procedure’ section to see a list of required items that students must include in their procedures prior to the approval of their procedure.

Day 3: During this lab period the students should create the resin itself. Students should follow the laboratory procedure that was pre-approved by the instructor the day prior. Students should ensure that they follow all relevant safety precautions, and be especially careful around the hot plates, oil baths, and while handing the hot resin.

Day 4: During this shorter class period students will use their resin to create dog bones for testing. Similar to the previous day, students should follow the laboratory procedure that was pre-approved by the instructor. On this day, students should take precautions not to touch the UV lamp, nor look directly at the lamp.

Day 5: During this shorter class period students will set up the apparatus they will use to test the force (either maximum or force at break) that their dog bones can handle. Students should try to pull the dog bones with the force sensor at a slow, and constant rate as possible.

Day 6: Students will have this final lab period to analyze their results and create a final project poster. The instructor may choose to provide guidelines for students to follow while creating their final project poster using the sample rubric listed on the next page.

Final Project Sample Rubric:

Standard	Advanced (2)	Proficient (1)	Basic (0)
Background Information	Background section thoroughly explains the relevant concepts (polymerization, esterification, and Young's modulus).	Background section touches on all concepts but is not thorough OR does not touch on all relevant concepts.	Background section is incomplete, with multiple concepts missing.
Materials and Methods	Materials list includes all materials used in the lab AND Methods section is a thorough description of the experiment that was performed.	Materials list omits a few (1-3) materials used in lab OR Methods section omits a few (1-3) procedural steps.	Materials AND Methods are incomplete, missing many materials used in lab or procedural steps taken.
Results	Results tables/graphs contain proper formatting, including axes, units and graph titles. Results section contains all relevant data collected from the lab.	Results tables/graphs contain minor formatting errors or omissions (1-3 per table/graph) OR Results section does not contain all relevant data collected in the lab.	Results tables/graphs contain a large number of errors or omissions AND Results section does not contain all relevant data collected in the lab.
Analysis	All graphs/tables are clearly explained. Connections are made between the results and background information.	Tables/graphs are not clearly explained OR no connections are made between the results and the background information.	Tables/graphs are not clearly explained AND no connections are made between the results and the background information.
Future Work	A thorough explanation of future work is proposed, justified from the background information and the experiment results.	Future work is proposed, though the justification may be incomplete, missing, or not supported given the results of the data.	Future work is not proposed, or work proposed is not related to what was done in the lab.
Layout	The poster layout follows a clear logical pattern, the text and tables/graphs are easy to read, and the appearance of the poster is neat and organized.	The poster layout satisfies two of the three criteria (clear logical pattern, legible texts and tables/graphs, and neat and organized appearance).	The poster layout satisfies one or none of the three criteria (clear logical pattern, legible texts and tables/graphs, and neat and organized appearance).

A grading scale that accompanies this rubric is posted on the next page.

Grading Scale:

Standard Grade	Number of Points	Percent Scored
Advanced	11-12	100%

Approaching Advanced	9-10	93%
Proficient	7-8	85%
Approaching Proficient	5-6	75%
Basic	3-4	65%
Below Basic	0-2	45%

Lab Procedure

The students will create the procedure for the lab. The instructor may choose for the students to create two or three separate procedures, for each part of the lab. It is advised that students should create at least two separate procedures, one for the creation of the resin and creation of the dog bones, and one for force testing of the dog bones. Regardless of the number procedures chosen, the students' procedures should contain at least the following, in this order:

- Massing of reactants
- Setup of the reaction apparatus (round-bottom flask in an oil bath)
- Creation of the prepolymer
- Formulation of resin
- Preparation and curation of the polymer dog bones
- Force testing

Similarly, as students create a procedure, they should likewise have prepared one or multiple data tables (teacher discretion). Their data tables should ensure they collect at least the following information:

- Any safety concerns associated with the reactants chosen, hot plates, oil baths, and instrumentation required for force testing.
- Mass of reactants
- Qualitative data about massing the reactants, resin setup and creation.
- Qualitative data about the creation and curing of dog bones.
- How long each of the dog bones were cured for (time).
- The dimensions of the dog bones.
- Force data (the instructor can choose whether to require students to collect the maximum force, and/or force at break, or have students propose what data to collect).

Lab Cleanup

The resin will be sticky! Ensure that gloves are used to prevent the resin from touching your skin or clothes. Extra resin can be thrown into the trash.
The oil baths can be reused from class to class and saved for future years' experiments.
Beakers should be washed using acetone or IPA, then with water, to remove all possible resin.
Dog bone fragments can likewise be thrown into the trash after experimentation.