**March 21, 2018 Lab Information**

**BIEN 235 Applied Biomaterials**

**Polymer Synthesis Lab**

In this lab, you will divide into teams of 3-4 students and you will make three polymers. The polymers include a pliable hydrogel, a rubbery polymer (elastomer), and a relatively rigid polymer. You will do a short set of experiments with the hydrogel. All polymers will be saved for tests in subsequent labs. Write notes on the instructor’s discussion and your observations during the lab; there will be several homework questions on the polymer synthesis and properties.

**Safety**

Place any food and beverage bottles inside your backpack (away from your work area) or on the table designated for safe storage. Wear goggles and nitrile gloves. You were instructed to bring your own goggles. We will supply gloves. Also, wear closed shoes and long pants. Please don’t spill solutions because we only have a small amount of materials for each class and we cannot replace what is lost. If you spill a solution on the table or floor, wipe it up with paper towels and place them in a trash can with a plastic liner. If you spill irritating solutions (borax, methyl methacrylate liquid, Sylgard base and activator) on yourself, remove your lab coat and tell the TA or instructor (who will put it aside for washing). You can wash off small spills on your hands/arms in the restroom. If you get a lot on your clothes (soaks through lab coat), we will take you to the safety shower. Also, read the specific safety data sheet (SDS or MSDS) instructions for each material (posted in the classroom). If you have any questions, please ask the instructor or teaching assistant.

**Labeling your team’s polymers**

Each team will have three paper cups, one for each polymer. Using a permanent marker, write at least one team member’s name on the cup, the date and the name of the polymer. You will use these in upcoming lab exercises.

**Hydrogel Synthesis**

**Preparations before class**

Preparation of solutions *will be* *done by the class instructor or teaching assistant*.

**Materials**

Two materials will be mixed to form a hydrogel:

1. Aqueous solution of polyvinyl alcohol (PVA), 4% (vol./vol.). The chemical formula for vinyl alcohol (the “mer”) is -[CH2CH(OH)]*n*-. The “*n*” in the formula is the average number of mers in a chain. Our PVA has an average molecular weight of 150,000 g/mol.
2. Aqueous solution of sodium borate, 4% (vol./vol.). Na2B4O7 ·10H2O is the chemical formula of the powder. When in solution, it forms borate ions. Borate has one boron atom surrounded by 4 hydroxyl (OH) groups. Borate ions bonds with the OH groups in the PVA chains which crosslinks PVA chains. This forms a porous 3-dimensional material that holds water.

**Safety**

Wear PPE described above. The sodium borate solution (borax) is used in detergents and can irritate the skin, eyes and mucous membranes if a concentrated solution is not washed off. See the SDS, placed on the table at the work station, for details. Once the borax is thoroughly mixed with PVA, the crosslinked polymer can be handled without gloves. However, you should wash your hands before eating.

**Synthesis**

1. Put on a lab coat (supplied) while measuring and stirring materials. Also, wear your goggles and gloves (supplied).
2. Use the measuring cup provided to transfer the polyvinyl alcohol (PVA) solution to plastic bowl.
3. Use a measuring cup (provided) to add warm water to the PVA solution.
4. Quickly add and stir the borate solution. If the borate solution cools, crystals fall out of solution, and the hydrogel won’t gel correctly.
5. Remove the hydrogel from the PVA cup with gloved hands and knead it thoroughly to finish mixing the contents. Roll the hydrogel around in your hand, gently squeezing the material to remove air bubbles at the same time. Alternatively, place the hydrogel into a plastic Zip-LockTM bag and repeatedly squeeze the mixture from outside the bag.
6. Once all the liquid becomes a gel, you can handle the polymer without gloves, if you wish. If so, you should wash your hands after the lab or before you eat. Be sure to conduct the tests, below.
7. After performing the four tests below, place the gel into a cup pre-labelled with “PVA,” the date, and the name of one of your team members. Put the cup into a sealed plastic bag (e.g., a Zip-LockTM bag).

**Test the properties of the hydrogel in the following ways**.

Write notes on what happens in your lab notebook after doing tests 1 – 4, below.

1. Pull the hydrogel apart slowly.
2. Pull the hydrogel apart sharply and quickly.
3. Roll the hydrogel into a ball, place it on the table and hit it hard with your hand.
4. Roll the hydrogel into a ball, place it on the table and wait 5 minutes. Observe the behavior of the gel. To save time, this step can be done by one person after everyone on the team has done procedures 1 – 3.
5. Remember to place your hydrogel in a labeled paper cup and then put the cup in a sealed plastic bag.

Although only one or two people on your team probably got to make the hydrogel, each team member must perform the first 3 tests above and write notes on what happens during the 4 tests. The tests are designed to illustrate the viscoelastic nature of polymers without the need for highly specialized equipment that would be required for other, more rigid polymers. You will be asked several questions about the behavior of the gel on your next assignment; so, it would be a good idea to take notes or a video.

**Polydimethylsiloxane (PDMS) Synthesis**

**Materials**

This elastomer is made from a viscous solution of long chains of of polydimethylsiloxane and a liquid activator with short PDMS chains. The powder comes in a plastic bottle and the activator is in a squeeze bottle. These are supplied as a kit from Dow Corning called Sylgard 184.

**Safety**

Wear PPE and follow instructions, as described in the first Safety section above. Read the SDS for the two materials in this kit.

**Synthesis**

The base and activator are mixed in a 10 to 1 ratio.

1. Scoop 1 *tablespoon* (5 ml) of Sylgard 184 Base into a paper or plastic cup labelled with “PDMS,” the date, and the name of one of your team members. The solution is viscous and will take time to pour. Place the measuring spoon on the paper towel by the container when finished.
2. Use the “Dash” measuring spoon to place a small amount of activator in the cup with the base. Be careful not to get the base solution on the spoon. If this happens wipe off with a paper towel and put the towel in the black trash can. Place the measuring spoon on the paper towel provided next to the container when finished. (It will be sticky.)
3. Go back to your work station and use a wooden stir stick to mix the two solutions. Care should be taken to minimize air entrapment. It is best to keep the end of the stick on the bottom of the cup while moving back and forth along the bottom of the cup. Cover the entire bottom area using this method to ensure thorough mixing.
4. Place your cup in the designated storage area in the lab to cure.

**Pot Life and Cure Rate**

The crosslink reaction (“cure”) begins with the mixing process a there is a gradual increase in viscosity, followed by gelation and eventual conversion into a solid elastomer. The material “pot life” is the time needed for the viscosity to double after the powder base and activation (or curing) agent are mixed. The pot life is highly temperature and application dependent. The polymer will take a couple of days to fully cure at room temperature (25°C). For future reference, the curing time can be reduced by heating the mixture. The manufacturer supplies a table (copied below).

|  |  |  |  |
| --- | --- | --- | --- |
| **Cure Time at 25ºC** | **hours** | **48** |  |
| Heat Cure Time at 100ºC | minutes | 35 |  |
| Heat Cure Time at 125ºC | minutes | 20 |  |
| Heat Cure Time at 150ºC | minutes | 10 |  |

**Temperature Ranges for Use**

Copied from Dow Corning Sylgard 184 Product Information Sheet. “For most uses, silicone elastomers should be operational over a temperature range of -45 to 200°C (-49 to 392°F) for long periods of time. However, at both the low and high temperature ends of the spectrum, behavior of the materials and performance in particular applications can become more complex and require additional considerations and should be adequately tested for the particular end-use environment. For low-temperature performance, thermal cycling to conditions such as -55°C (-67°F) may be possible, but performance should be verified for your parts or assemblies. Factors that may influence performance are configuration and stress sensitivity of components, cooling rates and hold times, and prior temperature history. At the high-temperature end, the durability of the cured silicone elastomer is time and temperature dependent. As expected, the higher the temperature, the shorter the time the material will remain useable.”

*Source:* Dow Corning Product Information Sheet, Form No. 11-3184B-01, April 2, 2014

**Testing your polymer**

You will test some of the properties of your PDMS in a future lab.

**Polymethyl methacrylate (PMMA) Synthesis**

**Materials**

This polymer is made from a fine powder of polymethylmethacrylate (short chains of PMMA) and a monomer, methyl methacrylate. The powder comes in a plastic bottle and the activator is in a squeeze bottle. These are supplied as a bone cement kit from DePuy. DePuy donated a few different kits that have a variety of cure times and temperatures.

**Safety**

Wear PPE and follow instructions, as described in the first Safety section above. Read the SDS for the two materials in this kit.

**Synthesis**

The activator liquid smells and prolonged exposure to fumes could make some people feel ill. So, you must mix this in a fume hood. We will use the fume hood in the histology lab (this was one stop during our lab tour last week.)

1. Measure 5 1 level *tablespoon* of powder into a paper or plastic cup pre-labelled “PMMA” with the date and the name of one of your team members.
2. Add about 1 ml (~1/2 *teaspoon)* of methyl methacrylate (liquid activator) to the powder.
3. Mix thoroughly with a wooden stir stick.
4. Put the cup in the back of the hood to cure. This will take about 5-10 minutes depending on the amount of liquid activator that was used. After curing and evaporation of the liquid activator, it can be removed from the hood and passed around to lab members to see to polymer. It will till have a smell, so put it back in the hood for storage.

**Pot Life and Cure Rate**

The crosslink reaction (“cure”) begins with the mixing process a there is a gradual increase in viscosity, followed by solidification. The material “pot life” is the time needed for the viscosity to double after the powder base and activation (or curing) agent are mixed. The pot life for PMMA is a few minutes and is highly dependent on the amount of activator that is added.

**PMMA Use**

PMMA has been used as a dental cement, bone cement (our DePuy kits), denture material, and more. It is naturally translucent, but colors can be added, such as when it is used for making dentures.

Last update 12/12/2018