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**Contact: Prof. Bassam Z. Shakhashiri**

**UW-Madison Department of Chemistry**

**bassam@chem.wisc.edu**

**www.scifun.org**

# From Molecules to Models:
# Advances in the 3D Printing of Polymeric Materials Toward Improved Control, Functionality, and Sustainability

By

Kyle C. H. Chin

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The dissertation is approved by the following members of the Final Oral Committee:

Andrew J. Boydston, Faculty, Professor, Chemistry

Padma Gopalan, Faculty, Professor, Material Science and Engineering

Reid Van Lehn, Faculty, Associate Professor, Chemical and Biological Engineering

David Lynn, Faculty, Professor, Chemical and Biological Engineering

# Chapter 12: Thesis for the Layperson

Thank you to the Wisconsin Initiative for Science Literacy, specifically Cayce Osborne, Elizabeth Reynolds, and Bassam Shakhashiri, for this opportunity to explain my thesis work to the public. I believe scientific literacy is important on multiple levels:

- From an **individual** level, leveraging professional expertise allows one to make informed decisions that could be crucial to your health or wellbeing.
- From an **educator** level, understanding scientific literacy and improving science communication can break down barriers in education.
- From a **professional** level, scientific literacy enables you to connect your expertise to others' expertise. Notably, an expert in one area is often a layperson in another, nonetheless the connection between those two areas could have huge implications.
- On a **societal** level, the public often funds much of the research being done and scientists have an obligation to justify the funds. Additionally, at some point we hope non-scientists will be able to use the results of our research in some meaningful way and people should have the option to understand how it works.
- From a **personal** level, writing this chapter prepares me for future effective collaboration. Additionally, during my PhD, in conversation with fellow graduate students, someone said, "I feel like I know less now than when I started". Of course, this is not meant literally, but represents how disconnected you can get from what is general knowledge and what is specialized. As you dive deeper into your field, more questions arise than you can answer, and so by writing this chapter, hopefully, I can appreciate how far I have come.

### Q: What will this chapter cover?

Throughout my thesis work, I have delved into the world of polymer materials and 3D printing (more on these in a bit!). Specifically, I examined how we can design materials for 3D printing with both improved properties (for example, stronger materials), new functions (for example, materials that respond to their environment), and better sustainability. This chapter is for anyone in the public who is interested in learning more about how scientists think about designing new materials. In contrast to the other sections of this thesis, this chapter will be limited in technical jargon or any assumptions about the reader's prior knowledge. Through this chapter, I will answer the following questions:

1. *What is the role of a material scientist?*
2. *How do material scientists go about designing new materials?*
3. *What is a polymer?*
4. *What is 3D printing and how can one 3D print a polymer?*
5. *What are some materials I made during my PhD?*

Hopefully, by the end of this chapter you understand a bit more about the motivation behind developing new materials and how I and other material scientists think about designing new materials. I hope you enjoy learning about my PhD work!

## Q: What is the role of a material scientist?

Materials have been extremely important throughout human history. So important, that entire time periods have been described by their defining materials. Some examples are the stone age and metal age, which each brought unprecedented changes to civilizations and technology. In fact, even the present is defined by two materials—plastics (found everywhere from water bottles to clothing to medicines) and silicon (used in computers and other electronic devices). While materials have substantially evolved from those materials that can naturally be found, such as wood and stone, to synthetic materials, such as steel and Kevlar, the material scientists' role is never done. Material scientists are constantly developing materials with ever improving properties to solve some of the world's most pressing problems, such as clean and renewable energy, sustainability, space travel, human health, computing, and more.

When I came to graduate school, I decided I wanted to learn how to design better materials. While I was not entirely sure what materials I would design, I knew I wanted to understand how to transfer fundamental understanding of physics and chemistry to desired properties and functions. Early on in my graduate school coursework, I came to understand that better control of a material's structure was key to achieving this, and that 3D printing was a revolutionary technology. I also came to the realization that polymers are an important class of materials that offer huge opportunities for developing novel functionality, but also have a pressing need to improve their sustainability. For the last five years, I have been exploring these aspects, growing as a material scientist, and learning to tackle pressing challenges that could be solved by developing new materials. I hope you enjoy reading about what I learned and some of the materials that I developed.

**Q: How do material scientists go about designing new materials?**

Aspiring material scientists learn early on the importance of the relationship between a material's structure, processing, property, and performance (Figure 12.1). Importantly, a fundamental lesson is that each of these elements are interconnected, and that by changing one, you affect another. While this can complicate material design, it also creates a vast number of variables that can be changed, yielding enormous room for growth and discovery. However, it also makes it clear that the more control we have over this design space, the more we can learn and improve.

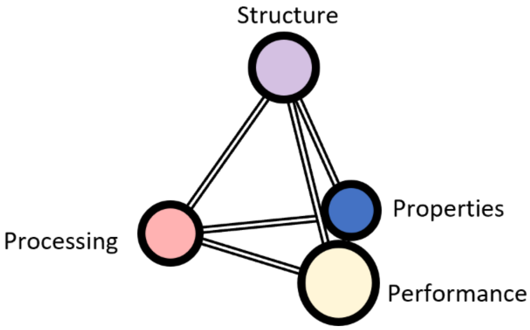

***Figure 12.1.*** *Material science tetrahedron showing the relationship between structure, processing, properties, and performance.*

One way material scientists can improve materials is through controlling the structure of a material across length scales: from the molecular to the macroscopic. So, what does this mean? Well imagine you want to make a Lego™ model. When you dump out the pieces you would likely see they come in many shapes, sizes, and connectivity. If you connect each piece in the precise location with compatible pieces one at a time, perhaps building smaller subunits first before assembling them together, you will eventually form the desired final structure. Depending on how these pieces are connected your final structure can take on very different properties. For example, maybe it can fold, be quite rigid, be easy or difficult to be unassembled, or have room to expand on in the future. Taking a step back, you can see that you have used the structure and properties of your smaller individual building blocks to create a larger structure with new properties, shape, and function. Material scientists attempt to do this with similar levels of precision, but with building blocks so small they are impossible to see or touch. So how do we do this?

Well first, we need to put into context material scientists' building blocks, atoms. Atoms are the basic building blocks of all the matter around us and compose everything we can see, touch, and smell. Each atom is composed of three subatomic particles called the proton, neutron, and electron (Figure 12.2A). The proton and neutrons are housed in the nucleus of the atom, while the electrons fill the volume around the nucleus. For simplicity, one could envision it being like the way planets orbit the sun (Figure 12.2B). The sun, in this case, is the nucleus, while the planets are electrons. However, the entire size of an atom is around an Angstroms (Å), which is 10 billion times smaller than a meter. Notably, an atom can have different numbers of protons, neutrons, and electrons, and a single type of atom is known as an element. All the known elements are represented on the periodic table. Therefore, the periodic table contains all the building blocks material scientists can use to design materials. Some of the elements you have likely heard of before, for example hydrogen, oxygen, nitrogen, copper, and gold. Notably, just like Lego™ blocks, elements come in many different shapes, sizes, and connectivity, which controls how they will interact with atoms identical to themselves or other elements in the periodic table.

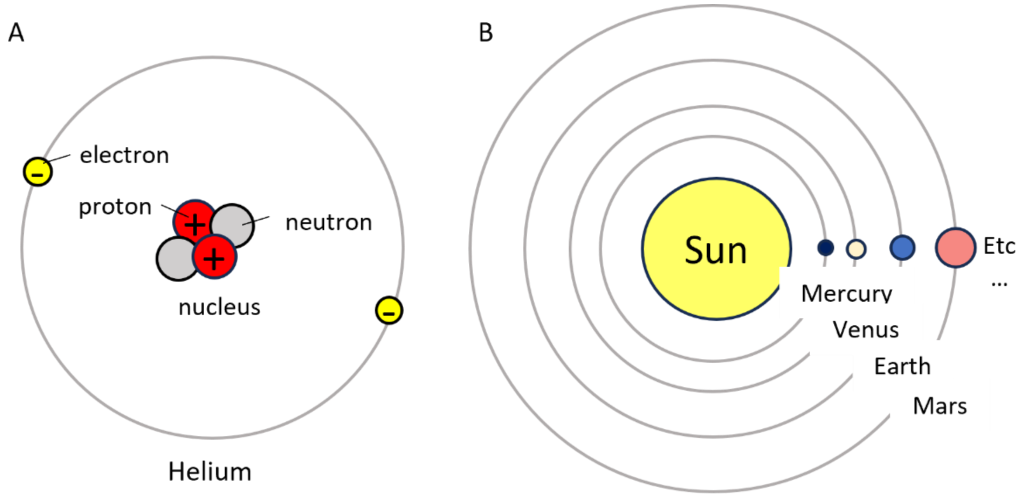

***Figure 12.2.*** *A) Picture of an atom of the element helium (He) B) General diagram of planets orbiting the sun.*

In reality, not many pure elements are found in nature, but rather elements are commonly assembled into structures called molecules. A chemist's job is to figure out ways to take elements or molecules and react them with one another to form new useful molecules. For example, two hydrogen atoms (H) and one oxygen atom (O) can react to form water ($H_2O$) (Figure 12.3). Notably, molecules can also react with one another to form new molecules, which will become important later in this chapter. While there are rules for how molecules react with one another, this is quite complicated, and so for now, understanding that we can make a wide variety of molecules made up of atoms is key!

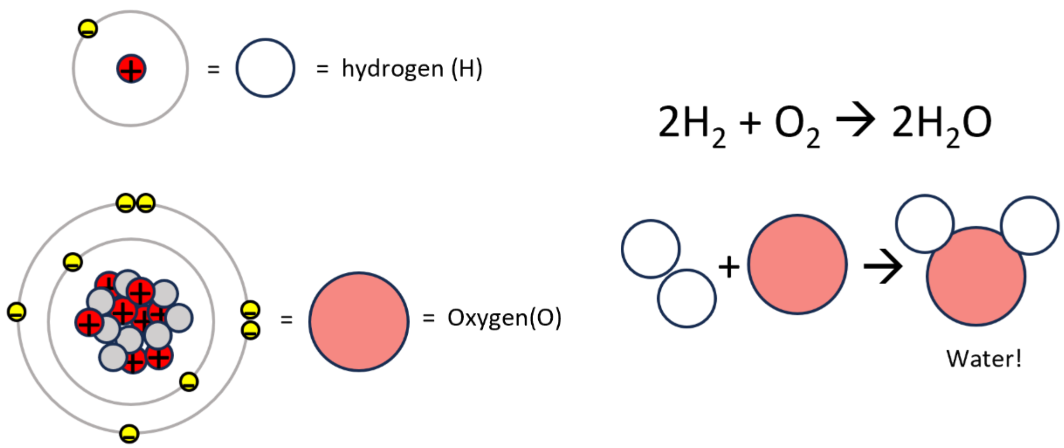

***Figure 12.3.*** *Diagram of oxygen, hydrogen, and the water molecules as well as the general reaction of hydrogen and oxygen to form water.*

With atoms and molecules, we can do quite a lot. By controlling how they are arranged in space, we can form larger structures that we can begin to see and manipulate (Figure 12.4). For example, we can take many water molecules from our faucets and fill a glass with them. In the glass, the individual water molecules are around one another in a disordered fashion, and can move relatively freely. Because of this, the molecules make a liquid that we can readily pour into a new glass, or ice mold. However, by altering how we process the water, its properties can change drastically. For example, when we sufficiently cool water down it freezes, the molecules become ordered and get locked into place, losing mobility, and the water becomes solid ice. Suddenly it has become quite hard, and we can no longer smoothly pour it. Additionally, if we heat the ice up, the water will return to its liquid state. If we continue to heat it up, it may suddenly begin to boil into gaseous steam. Now, we can no longer contain it in a glass, but we can use it to power things, like trains, using steam engines. This illustrates the drastic effects that the processing of a material has on its physical properties.

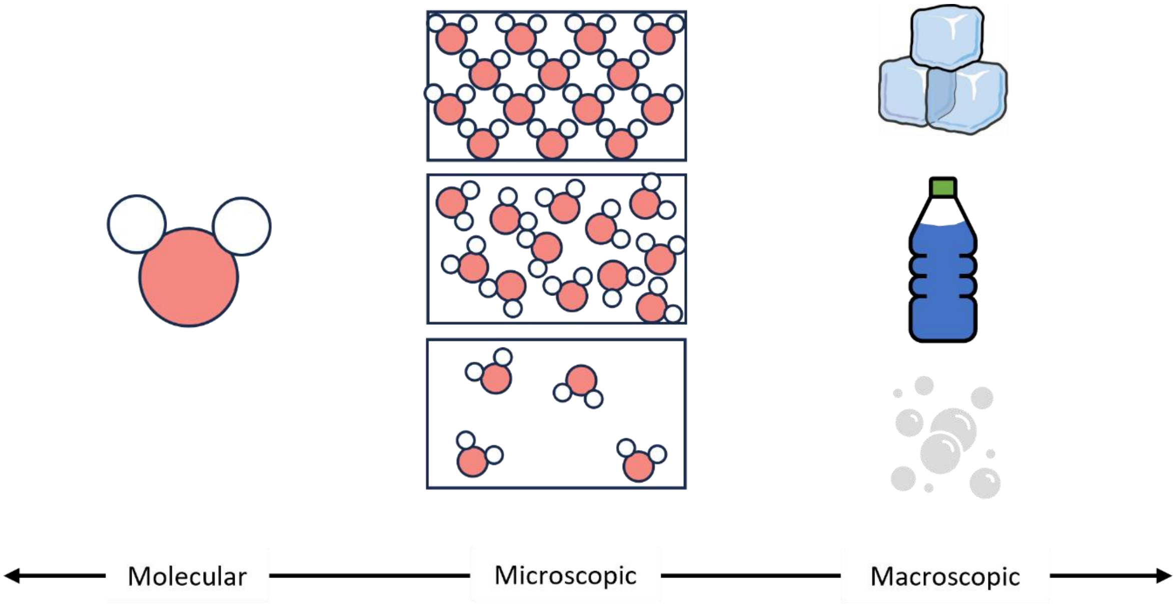

***Figure 12.4.*** *Visualization of water at different length scales. This illustrates that the way water is structured on the molecular and microscopic level has a profound effect on water on the macroscale.*

In summary, material scientists use structure and processing to control the properties and final performance of a material. To do so, they use building blocks, called atoms, and structures made of atoms, called molecules, to build useful materials. New materials can be made by reacting different atoms or molecules together to form new molecules. Notably, even a single material's properties can vary dramatically depending on how it is processed. Now that we have this level of understanding, I will narrow the scope to a single class of material that we can make with molecules, called a polymer, which is the focus of the next section.

**Q: What is a polymer?**

Polymers, composition of the Greek words *poly* and *meros*, meaning many parts, are large molecules (aptly termed macromolecules) composed of many repeating sub units called monomers. These macromolecules can simply be thought of as a paper clip chain where each individual paper clip is a monomer, and the entire chain is the polymer (Figure 12.5). The process of forming a polymer is called polymerization and entails linking monomers together to form a longer chain. Notably, polymers can be made from a single type of monomer, termed a homopolymer, or multiple types of monomers, called a copolymer. For the case of copolymers, they can be arranged into different patterns such as random, blocks, or alternating (Figure 12.5). It turns out different patterns can drastically alter the properties of the polymer.

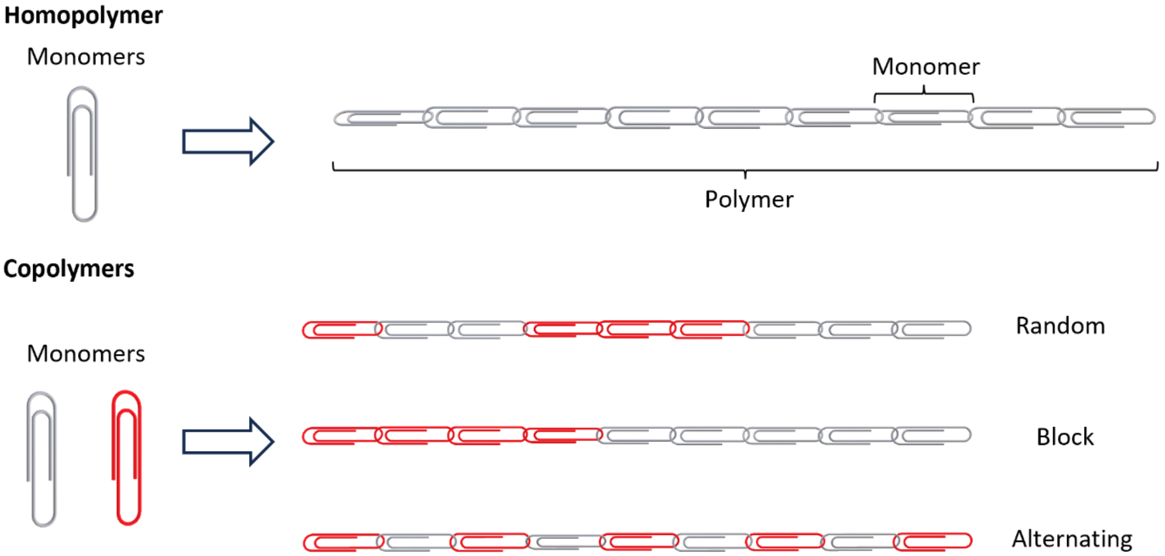

*Figure 12.5. Example of a polymerization reaction to form a homopolymer or a copolymer illustrated with paper clips.*

Polymers are found virtually all around us (Figure 12.6). This is because a polymer's macromolecular nature instills many properties that are advantageous, such as being light, tough, and chemically resistant. Frequently encountered polymers are materials such as polystyrene in packing peanuts, polyethylene in grocery bags, polypropylene in cups, acrylonitrile-butadiene-styrene in Lego™ bricks, polybutadiene in tires (rubber), and polyester in our clothing. Natural polymers are also all around us, such as cellulose in tree bark. Even our DNA is a polymer.

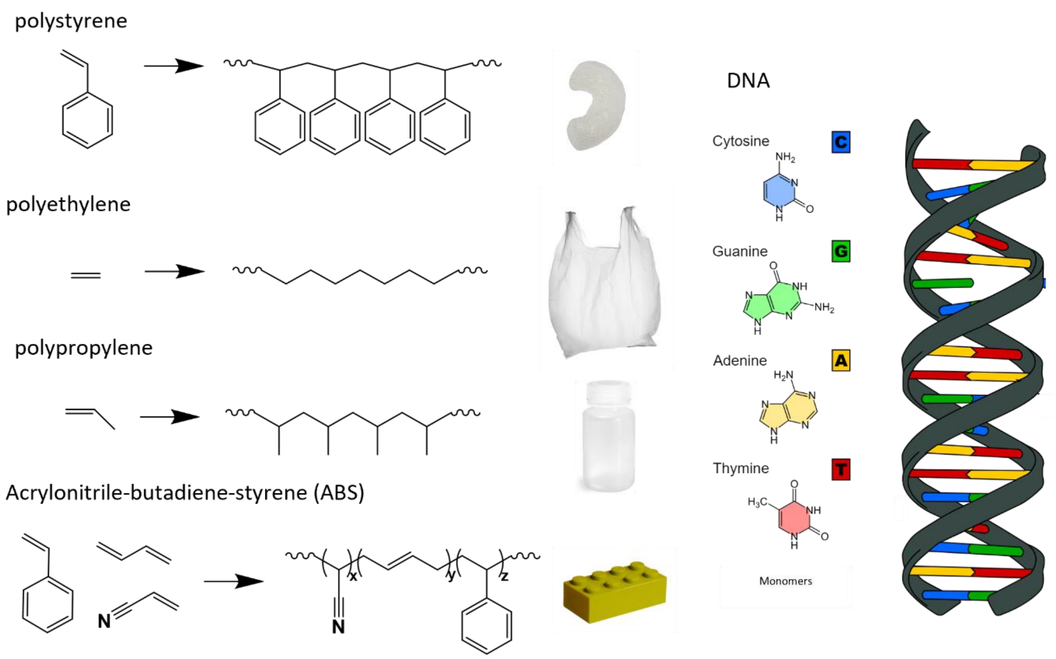

***Figure 12.6.*** *The formation of commonly encountered polymers from their monomers next to an example of where you would likely encounter that material.*

Synthetic polymers can be categorized in two categories, thermoplastics and thermosets. Thermoplastics are polymer chains entangled in one another; you can imagine a pile of paper clip chains. It might be difficult and take a while, but you can separate them all out into individual chains. Thermosets on the other hand, are polymer chains connected to one another with permanent bonds and form a fence like structure. You could imagine in the example of the paper clip chains, if I connect each chain to another by paper clip bridges. Now, the individual polymer chains cannot be separated into individual chains. On a molecular level, this has large implications, as the extra mobility of thermoplastics allows these materials to flow when heated, and dissolve when placed in compatible liquids, while thermosets cannot be reprocessed. This is important when we consider the future of polymers and improving their sustainability. For this reason, the most used thermoplastics are labeled with numbers that enable their sorting for reprocessing and recycling. On the other hand, tires are an example of a thermoset, unable to be reprocessed. Instead, they are ground up and often utilized as cushioning material in turf fields.

**Q: What is 3D printing and how can one 3D print a polymer?**

3D printing describes a process of manufacturing an object layer-by-layer from a 3-dimensional model constructed on a computer. There are many different types of 3D printing methods that span metals, ceramics, and polymers. Because objects are built in layers, we can produce complex structures that you would not otherwise be able to make, such as lattices, or customized objects specifically for an individual. This contrasts with more traditional methods of making objects, such as filling molds or sculpting. So far, this capability has been used to make customized products, such as dental implants, replacement organs, and foams used in football helmets and shoes (Figure 12.7). While 3D printing is still very much in development, it is being looked at as a revolutionary technology that could shift the manufacturing paradigm.

***Figure 12.7.*** *Showcase of applications of 3D printing technology and demonstration of the types of structures that can be formed.*

The main 3D printing technology I utilized in my thesis is called vat photopolymerization. Vat photopolymerization is a 3D printing process that takes a liquid and turns it into a solid using light (Figure 12.8). For this reason, we call the liquid a photoresin. Importantly, only the areas we expose to light will solidify. By doing this in thin layers (around the thickness of a human hair) and building layers one on top of the next, we can build up a fully 3-dimensional object. You might think this sounds like magic, but in fact it is polymer chemistry, and the reaction triggered by light is polymerization! You can think about this transformation like baking a cake. For a cake, you need certain ingredients that when mixed in the correct proportions make up your batter. After baking your batter in the oven at high temperatures, the liquid batter can react and solidify into a sugary treat. By baking many layers and stacking those layers, you can create an even taller structure.

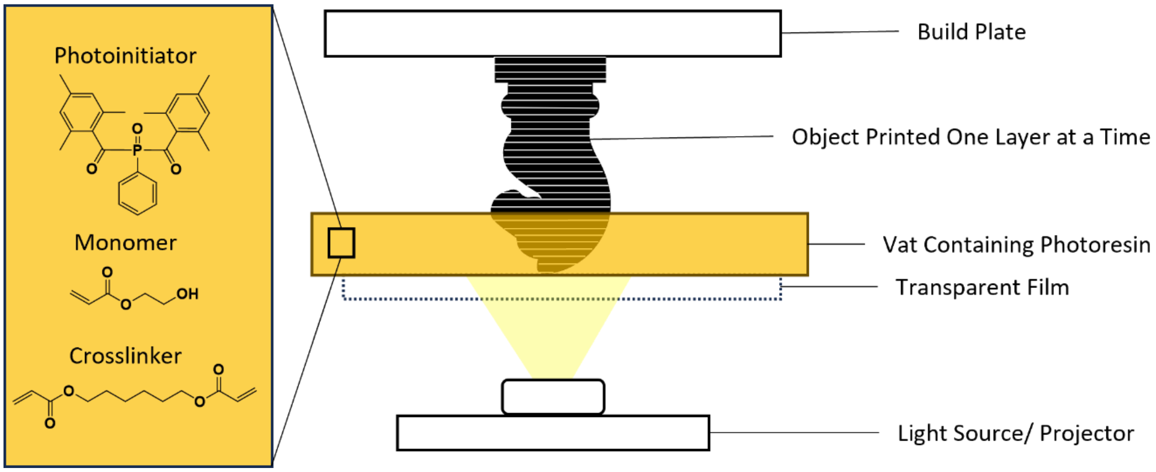

***Figure 12.8.*** *Schematic of a simplified vat photopolymerization printer with the different components labeled. Additionally, the basic ingredients to a photoresin are shown along with examples of what a specific ingredient might look like on a chemical level.*

Similarly, the ingredients (chemicals) and proportions (concentrations) of these ingredients I put into our photoresins determine how well the photoresin will print and what the final properties of the object will be. In the case of our photoresin, the three crucial ingredients are photoinitiator, monomer, and crosslinker (Figure 12.8). The photoinitiator is the chemical that reacts with the light and tells the polymerization reaction to start. The monomer is the major component to the photoresin, and will dictate the chemical structure of the polymer and therefore the material's properties. Lastly, the crosslinker enables the liquid to be converted into solid quickly. The more crosslinker you add, the faster the material will solidify, but often this comes at the cost of making your part more brittle. Importantly, adding a crosslinker turns the object that is being produced into a thermoset. Remember, thermosets are those materials that have a polymer chain connected into a fence like structure, meaning they typically cannot be reprocessed.

## What specific materials did I make during my PhD?

### 1. Tougher Materials

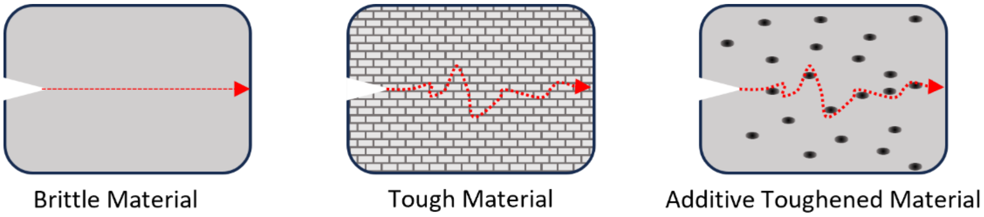

***Figure 12.9.*** *diagram showing the path crack of a brittle material, tough material, and a brittle material reinforced with a soft additive.*

A material breaks when a crack is formed and can propagate all the way through a material (Figure 12.9). One way to make a material less susceptible to breaking is by making the path the crack takes to reach the other side longer. A Common way that material scientists do this is by mixing the material you hope to toughen with soft additives. The role of these soft additives is to block the pathway of the crack, either by stopping the crack by absorbing its energy or by forcing it to take another pathway. However, these additives must be carefully designed, as they can also weaken the material by adding defects along the boundary between the additive and the material it is housed in.

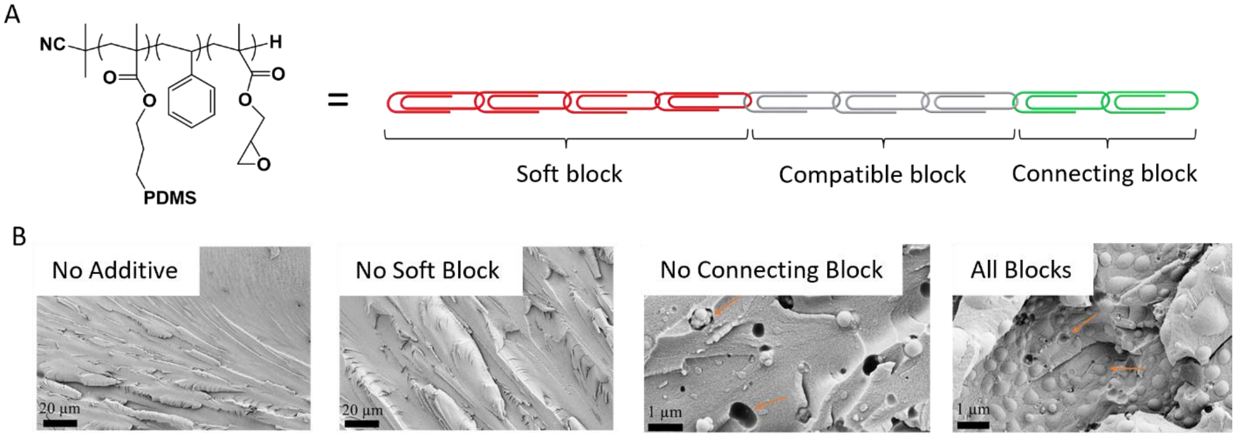

*Figure 12.10. A) Image of the exact polymer used in this study along with a diagram showing its triblock nature in terms of a paperclip copolymer B) Scanning electron microscope images of the bulk materials reinforced with polymers leaving out various blocks.*

During my PhD, I worked on a highly collaborative project combining the expertise of a polymer chemist, chemical engineer, and mechanical engineer. The project incorporated carefully designed polymers as an additive in a bulk material to improve its strength and toughness. Here, the strength of the material can be thought of as how much force it takes to break it, while the toughness is how much energy it takes to break it. The polymer was a triblock copolymer. The first block was soft and used to arrest and deflect a crack, the second block was used to make the polymer compatible with the bulk material, and the third block connected the bulk material to the polymer, ensuring it would not act as a defect. The molecular structure next to a paper clip model is shown in (Figure 12.10A).

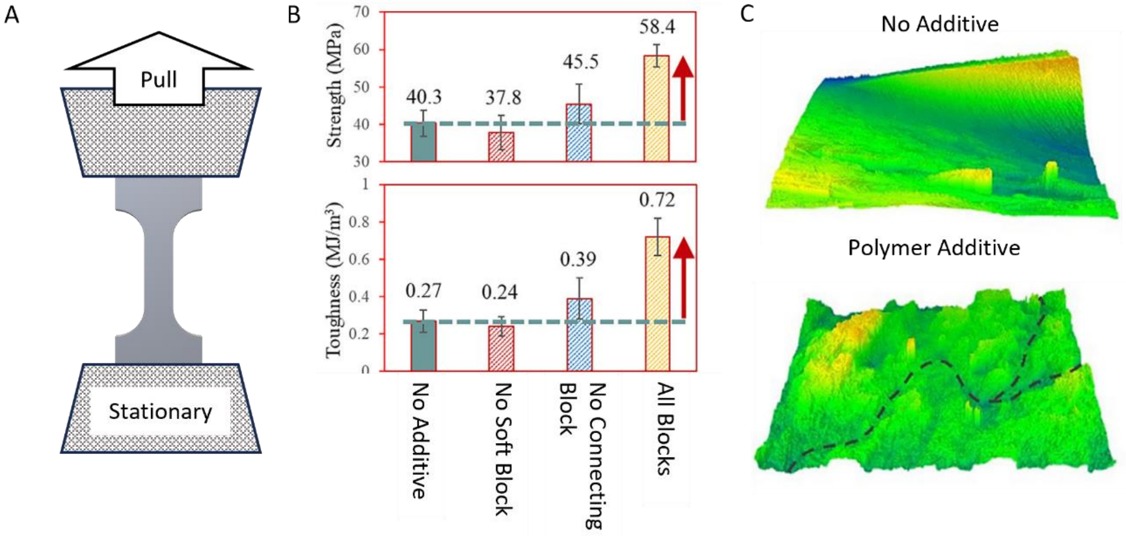

***Figure 12.11.*** *Schematic of the type of testing done on the materials, plot showing the strength and toughness of the materials with different polymer variations incorporated into the bulk material, and mapping of the fracture surface of a bulk material without additive and with polymer additive.*

To test this polymer, I 3D printed test samples consisting of a resin with the new triblock copolymer as an additive. Collaborators and I then looked at how it behaved in the bulk material. To do so, a collaborator who specialized in microscopy took pictures of the sample at high magnification to visually look at how the polymer arranges in the material (Figure 12.10B). We saw something very interesting. Firstly, without the polymer, the bulk material looks rather smooth. This did not change when we added a polymer with only the compatible and connecting blocks to the material. This means it is being cohesively incorporated into the material. However, when we added a polymer with a soft block into the resins, little pockets began to form within the material. These pockets form because the soft block is incompatible with the bulk material. Like oil and water, it does not like to mix, and so it tends to coil into a ball. However, these pockets are sometimes missing the polymer because it was not connected to the bulk material and came dislodged. When all blocks are present, we see the best effect: small pockets of soft material are housed and connected within the bulk material.

So does this lead to a stronger and tougher material? To test this, I printed samples that look like dog bones and pulled on them (Figure 12.11A), measuring the amount of force and energy required to pull the material apart, which indicates the material's strength and toughness. Using these additives, we were able to strengthen the material by ~1.5x and toughen the material by ~2x (Figure 12.11B). We saw no improvement in strength or toughness when there was no soft block, and marginal improvement without the connecting block. This shows us that all three blocks are needed for the best effect. We could also look at the surface of the broken sample where it cracked and map it (Figure 12.11C). You can clearly see that the sample with the additive is much rougher, indicative of the longer crack path during breaking.

## 2. More Sustainable Materials

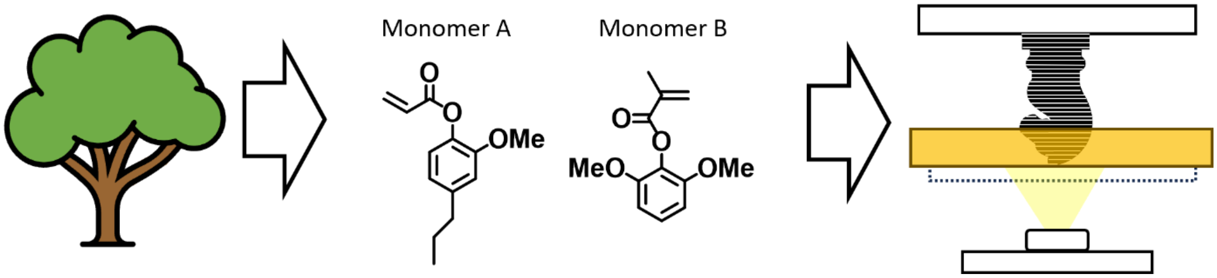

***Figure 12.12.*** *Diagram of the main idea behind the testing of more sustainable materials for 3D printing.*

Currently, vat photopolymerization resins are made from photoinitiators, monomers, and crosslinkers that are derived from petroleum feedstocks. As the world tries to reduce its carbon footprint, we want to move chemical production away from fossil fuels and toward more sustainable alternatives. One way to do this is to use biomass (materials from plants!). The most abundant biomass on the planet is that of lignocellulosic biomass (think of trees). Lignocellulosic biomass is made of three different polymers: cellulose, hemicellulose, and lignin. While cellulose components are used to make paper, the lignin component is often regarded as trash. This provides a huge opportunity, since we can now pull from a source that is already widely produced but not used. This is important, as increasing demand for biomass could lead to negative side effects, like deforestation.

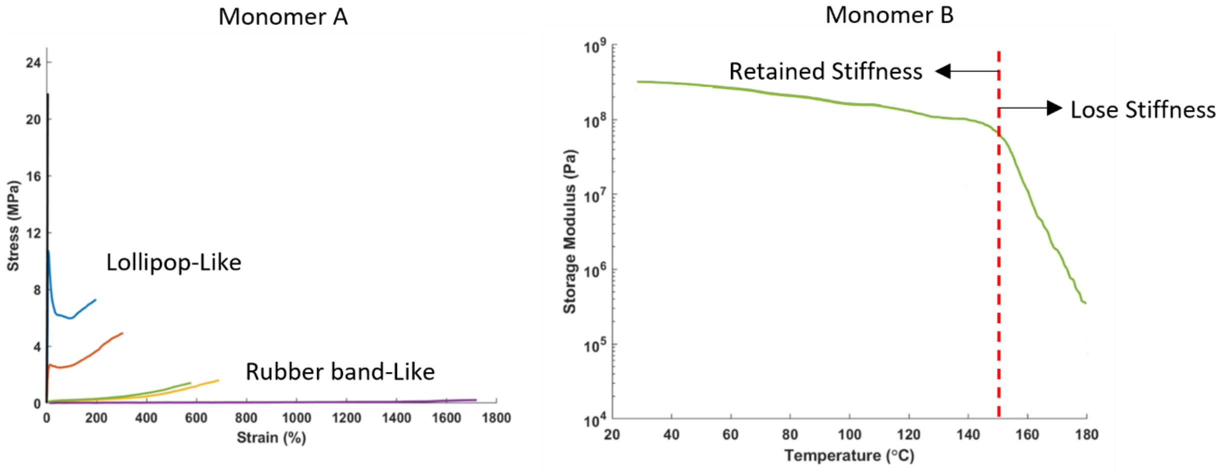

***Figure 12.13.*** *A) Mechanical response of different materials made from monomer A. B) Stiffness of a material made with monomer B with increasing temperature.*

To use lignin to make materials for 3D printing, lignin must first be deconstructed into usable smaller molecules. Many researchers are currently examining how lignin can be deconstructed, and how those different methods affect what molecules are produced. In my work, I looked at two different lignin derivable monomers (Figure 12.12), which I will call monomer A and monomer B, and examined those materials' properties, like strength, thermal properties, and how they react to different chemicals. Interestingly, I found that when I mixed monomer A with other bioderivable monomers, materials with highly tunable mechanical properties were produced. By stretching the different materials and measuring how far the samples stretched (also called strain) and how much force it takes to stretch (called stress), I observed highly varied responses, ranging from as stretchy as a rubber band to as stiff as a lollipop.

On the other hand, monomer B could be heated to over 100 °C (the temperature that water boils at) without softening. I tested this by measuring the storage modulus at increasing temperatures (12.13B). You can think of the storage modulus as how solid-like (or stiff) that material feels at a certain temperature. Typically, as you raise the temperature of a polymer, it will soften and become less solid-like, and this correlates with a drop in the storage modulus. Additionally, to print these materials, I did not need to add a crosslinker. This is uncommon, and enables printing of thermoplastic materials that can be reprocessed and recycled into new materials when placed into select liquids!

## 3. Stimuli Responsive Materials

***Figure 12.13.*** *diagram of a polymer with and without mechanophore illustrated by a rope with and without a fray. The fray indicates the idea behind incorporating a mechanophore into a polymer.*

When bone fractures, it can repair itself. Not only does it repair itself, but it can reconfigure, reinforcing those areas from future damage. Developing materials like bone would enable materials to adapt to fracture or cracking without any intervention from humans. Could you imagine a road that could fill its own potholes? Wouldn't that be great! One-way material scientists are beginning to think about this is by using mechanochemical transduction. This word may sound intimidating, but breaking this word down we have "mechano" meaning mechanical force, "chemical" meaning chemistry, and "transduction" meaning to convert. Therefore mechanochemical transduction is converting mechanical force into a chemical response.

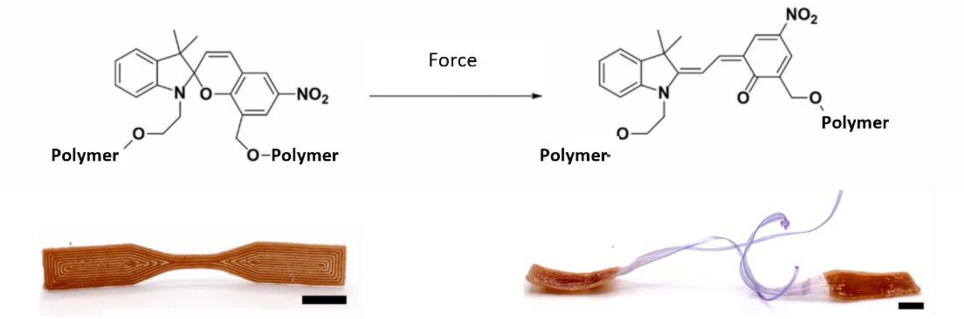

***Figure 12.14.*** *Example of a mechanophore used in our lab that changes color with force. At a molecular level the mechanophore called spiropyran changes its structure resulting in the color change.*

To do this, material scientists are designing polymers with mechanically sensitive units built into them. These mechanically sensitive components are called mechanophores. A simple way to think about these polymers is thinking about a game of tug of war. In a normal game, two teams will pull on a rope. If the teams are evenly matched, the rope will either stay where it is, or if they pull with enough force, the rope may even break near the center where the force was highest. However, what happens if we fray the rope in the middle? Now, when the teams pull, the rope will likely break at a much lower force than before at the exact location of the fray. This is essentially what a mechanophore does in a polymer. It acts as a weak point in the chain. However, this weak point can be designed to not only break the polymer chain, but also to change the structure of a molecule, which can cause a color change (as shown in Figure 12.14), or even release new chemicals.

## 4. Multimaterial 3D printing

Making an object from multiple materials can simply be done by mixing two materials together and then forming it into your desired shape. However, in this approach, you have no control over where each material will end up in the final structure. Making a multimaterial object, where you control the precise location of each material within the final object, is much more challenging. I have been approaching this problem utilizing a technique developed in our research group, where we use the color of light to form materials that have different properties. By controlling where we project different colors of light, we can then control the specific material that forms in that location. Importantly, this all occurs from a single photoresin!

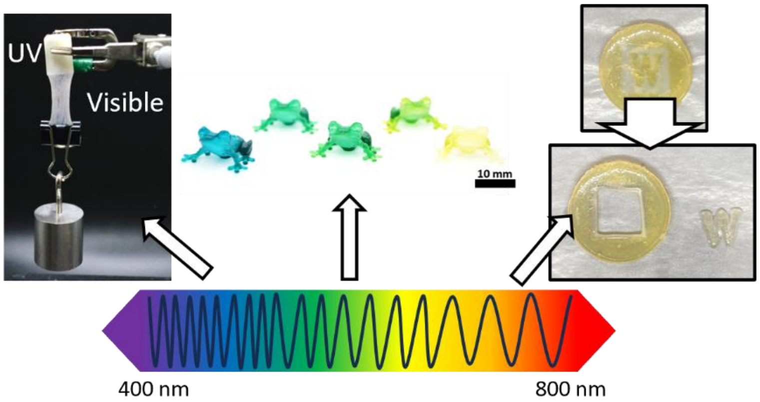

***Figure 12.15.*** *Examples of using different wavelengths of light to alter materials properties.*

This works by recognizing that different colors of light are each a different wavelength and contain different amounts of energy. Interestingly, molecules can be designed to respond to only specific wavelengths of light. This is similar to a radio. Each radio station is like a different wavelength of light. By tuning our radio to a specific channel, we can hear a range of content, including music, news, interviews, or sports. Our system works almost exactly like this, but rather than different content, we express different materials properties (Figure 12.15). For example, we are able to make materials that are soft with visible light and stiff with UV light. Additionally, I designed photoresins that can make materials that can dissolve with visible light, but that will not dissolve with UV light. Lastly, I developed a photoresin which uses visible light to form the object, and UV light to change its color.

## Summary

In conclusion, material scientists are important because they develop new materials to solve pressing problems in society. Material scientists do this by controlling the structure of a material across various length scales to achieve specific properties and performance. The building blocks used to do this are called atoms and molecules, which material scientists can design and carefully arrange into materials. One material that can be formed from atoms are polymers! Polymers are defined by the presence of large molecules with repeating units of monomers. These large molecules are lightweight and durable, making them very useful for creating many of the materials found all around us. One exciting avenue to explore using polymers is 3D printing. Using 3D printing, materials with complex structures can be made. During my PhD, I designed several polymer materials for 3D printing, including tougher materials, more sustainable materials, stimuli responsive materials, and precise multimaterials. Importantly, all of this was done by considering the properties of individual molecules used to make the materials.

## Where do we go from here?

Over the past five years, I have been able to design a range of polymers for 3D printing with improved strength and toughness, sustainability, stimuli responsiveness, and localized control over material properties. Through this experience, I have gained valuable insight into how to think about designing polymers from individual molecular building blocks, and how this affects their final properties and function. I hope I can use this understanding to better design the polymers of the future. From my perspective, the largest challenge facing the future of polymers comes from a sustainability standpoint. By 2050, the global production of plastics is forecasted to be 34 billion tons. This number, in combination with the growing plastic accumulation crisis and the formation of microplastics, necessitates solutions. Many of these solutions can come from better design of polymers at the molecular level, enabling improved biodegradability, recyclability, and a more circular plastic lifecycle.

To begin working on this problem, I will be joining a small team of material scientists and polymer chemists at RockyTech Ltd. in Boulder, Colorado. Our mission is to improve the nationwide recycling rate and simplify the current plastic recycling process. To do this, we will use a two-part strategy to upcycle existing plastics and develop novel polymer materials and composites with the material's entire lifecycle in mind. This enables us to better utilize the large accumulation of existing plastics while designing new plastics for the future.

## Image References

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