# FOOD SCIENCE COMMUNICATION: ENGAGING THE PUBLIC AND INTEGRATING SCIENCE COMMUNICATION IN ACADEMIC RESEARCH

### A Thesis

Presented to the Faculty of the Graduate School of Cornell University

In Partial Fulfillment of the Requirements for the Degree of

Master of Food Science

by
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August 2020



### **ABSTRACT**

Science Communication can be defined as any interaction or exchange of scientific knowledge. Engagement between scientists and non-experts can result in a better-informed public leading to better policy and consumer choices which is crucial to address high-impact issues such as climate change and public health, especially in the interdisciplinary field of food science where misinformation is commonly spread. My thesis describes a versatile and adaptable food science education program designed to not only educate the public, but also to train food science graduate students in science communication. Additionally, being an inherently collaborative field, food science communication between scientists is equally important as it promotes innovation and stronger research impacts that may also address solutions to climate change and public health issues. My thesis also presents a copolymerization method in detail, with specific protocol tips, notes, and reactor set-up diagrams.

### **BIOGRAPHICAL SKETCH**

Alexandra Macbeth started her undergraduate career at McGill University in 2014 where she majored in chemistry with a minor in mathematics. After two years, Alexandra transferred to New York University where she continued her major in chemistry. During her undergraduate career, Alexandra began academic research in the Sacanna Lab in the Molecular Design at NYU where she studied colloidal science. Her work focused on the synthesis of colloids into specific geometric shapes and their selfassembly into novel functional materials. In 2018, Alexandra Macbeth earned a Bachelor of Science in Chemistry Honor with a thesis on the synthesis of ring-shaped colloids intended for applications in liquid crystals. Alexandra went on to work in the food industry for KIND® Snacks as a laboratory technician on the New Product Development team where she built out the laboratory and implemented standard operating procedures. After a year, Alexandra began her master's degree in the Food Science & Technology program at Cornell University. There she conducted research in the Goddard Lab which focuses on biomaterials and biointerfaces. Alexandra's worked on protocol development and documentation for an emulsion polymerization to yield a metal-chelating, photocurable copolymer film intended for application in bioactive food packaging materials. Alexandra put a strong emphasis on science communication during her work which showed in her clear and concise methods paper General method for emulsion polymerization to yield functional terpolymers which is to be submitted for publication in Elsevier's journal, MethodsX. She also organized a food science event at Ithaca's Sciencenter with the intention of providing opportunities to train food science graduate students in food science communication and educate and engage the public in food science topics. After her master's degree, Alexandra intends to continue practicing and implementing science communication while pursuing a PhD in Chemistry at Cornell University.

I dedicate this thesis to Helen Hamaty (Sittoo) - Although she wanted me to be Mis
America, I think she would have settled for a scientist.

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### **ACKNOWLEDGMENTS**

I cannot begin to express my gratitude to Dr. Julie Goddard for her continued mentorship and guidance. The completion of this thesis would not have been possible without her input and support. I would also like to especially thank her for introducing me to Science Communication as a field and giving me the tools to pursue it. Dr. Bruce Lewenstein also played a special role in my immersion into Science Communication and I would like to extend a special thank you to him for supporting me in that. I would like to express my gratitude to my committee member, Geoffrey Coates, who has been a wonderful mentor and teacher as I continue to explore polymer chemistry.

The original food science education program was hosted at *The Sciencenter* in Ithaca, NY with special help from Coordinator, Lyla White and Executive Director Michelle Kortenaar. The graduate student volunteers from Cornell's Graduate Food Science Program were Rachel Carson, Catherine Dadmun, Devin Daeschel, Margaux Ehrlich, Brenna Flynn, Ian Kay, Sarah Kozak-Weaver, Timothy Lott, Meghan McGillin, Mariely Medina, Ann Van Le Nguyen, David Parker, Jessica Rafson, Elizabet Reyes, Autumn Rudlong, Jonathan Sogin, Zhixin Wang, Zirui Ray Xiong, and Mohammad Yaghoobi. This work was funded in part by the United States Department of Agriculture National Institute of Food and Agriculture under award # 2019-38420-28975, in part by Hatch under Accession Number 1014103 and in part by the Department of Food Science at Cornell University.

I thank Zhuangsheng Lin for his tremendous support in experimental design and modification.

I would also like to extend a special thank you to Autumn Rudlong and Hannah Zurier for assistance with the experiments and general help in the lab. The copolymerization methods

paper could not have been possible without characterization provided by the Cornell Center for Materials Research (CCMR) Facilities supported by the<GS5>National Science Foundation<GS5>under Award Number DMR-1719875. NMR analysis was conducted in the Cornell NMR facility with the technical assistance from Dr. Ivan Keresztes and Anthony Condo.

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### LIST OF ABBREVIATIONS

BA *n*-Butyl Acrylate

BPM 4-Benzoylphenyl Methacrylate

GMA Glycidyl Methacrylate

IA Itaconic Acid

IDA Iminodiacetic Acid

IM Itaconic Monoester

### **PREFACE**

There exists a knowledge gap between scientists and the non-expert public which is in-part caused by a discrepancy in science literacy. However, this disparity can be addressed through practice and implementation of science communication by those who have an extensive scientific background to those who may not. Science communication can most simply be defined as any interaction or exchange of science knowledge. In a world captivated by social media, it is becoming easier to broadcast information to large audiences which is equally advantageous as it is dangerous. Food science especially is a commonly misunderstood and miscommunicated field. The prevalence of food in our lives combined with its multi-disciplinary nature engenders discussion, but it is incredibly important that the topics discussed are scientifically accurate. Food science involves areas that are common concerns such as nutrition, food safety, food security, new food technologies especially as it relates to the clean label trend, agricultural sustainability, and climate change to name a few. Food is also a common source of enjoyment in people's lives as it fosters community, cultural exchange, and excitement. Many people are eager to understand and discuss the many facets of food science and it is up to those who are the most scientifically versed in these topics to facilitate these discussions to ensure the knowledge being communicated is factual. Proper science communication by scientists to the non-expert public leads to a better-informed public which in turn influences policy and consumer choices.

Science communication is equally important scientist-to-scientist, especially when it occurs across academic disciplines. As research becomes more interdisciplinary, new discoveries and knowledge must be transferred across fields, and more importantly must be *understood*. Whether it happens at conferences, in research meetings between departments, or even between lab group members, proper science

communication is crucial to accelerate research. Better science communication skills strengthen research impacts and lead to scientific advancement, especially in academia where students and faculty in the sciences are expected to teach, give talks about their research, apply for funding, and write publications on their work. Although there is clearly a need to train scientists to be skilled communicators, there exist few science communication training opportunities in universities. Thus, there is an urgency to implement science communication within academic curricula for both the faculty and student participation. Fortunately, there are many routes to take. For example, implementing public engagement programs that simultaneously educate non-experts while providing training opportunities for experts, or publishing more clear and concise research papers that provide step-by-step protocols to encourage collaboration and accelerate scientific breakthroughs. The former approach fosters scientist-to-nonscientist communication while the later encourages scientist-to-scientist communication, both of which are presented in the following thesis.

### CHAPTER 1

ENGAGED FOOD SCIENCE: DEVELOPING SCIENCE COMMUNICATION AT THE GRADUATE LEVEL WHILE BROADENING YOUNG SCIENTIFIC MINDS<sup>1</sup>

### 1.1 Abstract

Science communication is an essential tool in technology advancement, impacting both the communicator and the audience's understanding of scientific topics and issues. Without proper communication of ideas and knowledge in a way that is informative and accessible to a target audience, acceptance and implementation of new technologies can be hindered. Additionally, science communication is critical to increase public awareness about current scientific issues for more informed policy and consumer choices. Such skills are of critical need in food science, as many new food technologies are met with initial resistance by the consuming public. Building science communication skills of food science graduate students who are future leaders in their field (e.g. international agriculture, food safety, food security) is thus a critical, but often missing, part of their training. Here, we describe a food science education program designed so that graduate students can develop their science communication skills while also educating the public on topics in food science. This program consists of six activities that collectively introduce three areas in food science: food chemistry, food microbiology and process engineering. Here, we describe protocols for each activity

<sup>&</sup>lt;sup>1</sup> **Macbeth, A. J.**, Zurier, H. S., Atkins, E., Lewenstein, B., Nugen, S. R., Goddard, J. M., *Engaged Food Science: Developing Science Communication at the Graduate Level While Broadening Young Scientific Minds.* \*To be submitted for publication.

including a materials list, learning objectives and discussion points that are adaptable to different age groups, event spaces, or budgets. Each activity has a participatory component to ensure both audience member and instructor engagement. A program designed for food science communication not only inspires young scientific minds to better understand complex scientific topics, empowering them to envision a possible career in STEM fields, but also provides an exciting medium through which graduate students can practice and strengthen their science communication skills, benefiting not only their personal academic and professional skills, but also broader societal needs.

### 1.2 Introduction

It is increasingly clear that scientists must be able to communicate their research effectively to inform experts and non-specialist audiences to promote scientific advancement.<sup>1</sup> Furthermore, considering the lightening pace with which the media ecosystem is presently evolving,<sup>2,3</sup> misinformation can be spread by those who might be eager to share or understand, but do not have the adequate scientific background to do so accurately. Thus, proper science communication by scientists to non-scientists is critical to better inform the public and increase awareness about emerging scientific issues (e.g. environmental sustainability, new food technologies, and public health). Indeed, a better-informed public is a critical step in enacting policy changes or consumer choices.<sup>4</sup> Effective communication of new food technologies presents its own set of challenges in educating an often distrusting public to gain acceptability of new technologies designed to improve the quality, safety, and sustainability of our food system. The urgency and importance of improving food science communication is what

motivated the creation of a flexible and adaptable food science education program suitable for implementation in a range of outlets (e.g. science centers, public schools, science technology engineering and mathematics [STEM] engagement programming, community centers). Herein we present a versatile program designed for food science trainees to practice communication skills by informing the non-expert public of topics in food science. The science communicators, herein referred to as instructors, were food science graduate students from Cornell University who engaged with a public consisting of K-8th graders and their adult caregivers. There were six activities (located at individual tables throughout the science center), each with an experiential and interactive learning component, where graduate students conducted informal lessons in language accessible to both primary school students and their non-specialist adult caregivers, spanning three fundamental areas of food science: food chemistry, food microbiology and process engineering. Details of the event are reported in Table 1.1.

The overall objective of this program was to develop effective food science communication skills through engagement between the communicators and the public. *Science communication* has been defined as an interaction between scientists and members of the public regarding scientific topics,<sup>4</sup> while *engagement* is the exchange of science ideas between the communicators and the public, in other words, a mutually beneficial experience for both the learners and educators.<sup>5,6</sup> It was thus critical to provide these experiences in an environment conducive to effective learning. There is strong evidence that people of all ages can effectively learn science in informal

**Table 1.1: Event Details** 

Audience	Primary Audience: Students K-8th Grade
	Secondary Audience: Adult caregivers, Sciencenter staff/volunteers, Camp counselors
No. participants	Approximately 400
Chaperones	Adult caregivers
	Camp counselors
	Program directors
Participant	Advertisements by <i>The Sciencenter</i> and Cornell University's Department of Food Science.
recruitment	Intended for all students K-8 <sup>th</sup> grade in the greater Ithaca, NY area
Venue	The Sciencenter, Ithaca, NY
	Established 1983
	40,000 sq. ft.
	100,000 guests/year (Ithaca, NY)
	1.5 million guests/year worldwide via traveling exhibitions/public engagement programs
Duration	2 hr
Science	2 graduate students per activity table plus an additional graduate student to oversee the event
Communicators	logistics.
(a.k.a. Instructors)	
Subject Matters	Food Science: Food Chemistry, Food Microbiology (Food Safety), Food Process Engineering
	& Sensory Science
	Additional Science: Biochemistry (Proteins), Microbiology (Fermentation Science), Chemistry
	(Materials Science, Colloidal Science, Organic Chemistry, Polymer Chemistry, Emulsions &
	Emulsifiers), Physics (Emulsions, Dispersions)

settings.<sup>7</sup> People spend 95% of their lifetime outside of the classroom and are therefore more likely to encounter science in non-academic settings indicating that science communicators working in informal environments can reach audiences over a greater fraction of their lives.<sup>8</sup> Science museums provide an excellent setting for science communication and informal science education because they effectively bridge the gap between science communicators and the public.<sup>9</sup> As noted by Dr. Anissa Ramirez, scientist, author, and recognized expert in science communication, "Science museums are highly skilled at capturing the attention of young people. They do it all the time and they do it well." Therefore, while science communication can take place in many contexts, <sup>11</sup> this program was conducted in *Sciencenter*, a non-profit science museum in Ithaca, NY, enabling an engaging, non-intimidating setting for science communication and informal science education alike.

There is little distinction between science communication and science education but both are stimulated by science literacy. <sup>12</sup> Graduate students train to be scientifically literate in their field, in this case food science, but such depth of knowledge is less useful if not communicated effectively. Although graduate students often engage in science communication through teaching assistantships, presenting research at conferences to peers, through publications or when applying for external funding, graduate programs often do not include formal training for students to develop their science communication skills. <sup>4</sup> Providing more opportunities for graduate students to practice effective science communication, in this case using public engagement programs, will improve their impact, strengthen their research and aid in their professional development as scientists. Therefore, in this program, two food science graduate students were recruited for each

activity to effectively communicate and engage with the public and to grant them the opportunity to practice and strengthen their food science communication skills.

The field of food science is inherently multi-disciplinary, <sup>13</sup> integrating concepts of the physical sciences with which the general public have some degree of non-expert familiarity. Phase separation in salad dressings, the sensory experience of a fresh cucumber versus a dill pickle, or that of lactose free milk versus regular milk are familiar and relatable examples of food chemistry, food microbiology, and food process engineering & sensory science, respectively. While knowledge in these fields are complicated when presented on their own, given intentional context they can be much more comprehensible and accessible for any learner. Our program thus employed experiential learning, a pedagogical technique shown to improve comprehension of concepts that when presented alone might appear abstract, for participants of any age.<sup>14</sup> We also leveraged systems thinking, in which a relatable idea is used to describe a more complex phenomenon to improve student learning by introducing new information in context.<sup>15</sup> Herein, we report six experiential activities conducted at *The Sciencenter* in Ithaca, NY on February 19th, 2020 including for each the necessary materials and equipment, major learning outcomes, discussion points, and guiding questions for the instructor. Experiential learning and systems thinking techniques were leveraged in our what, why, how approach, in which the perceivable change was presented first as the what, followed by an introduction to the scientific principles that explain how and why that change occurs. These activities are designed for subsequent use either individually or as part of a larger experience as initially performed.

### 1.3 Methods

Each activity had the same overall objectives: 1) to grow science communication skills of food science graduate students; 2) to introduce scientific topics to an audience of students ages K-8<sup>th</sup> grade and their adult caregivers using experiential learning and systems thinking approaches. The learning outcomes for audience members were identified while planning each activity since science communication is most effective when the intended outcomes are predetermined.<sup>11</sup> Activity learning outcomes were defined based on The 2018 Guidelines for Initial IFT Approval of Undergraduate Food Science and Food Technology Programs and are included in the individual activities below and are unique for each activity. 16 However, the overarching goal for graduate students was for them to improve their science communication skills and thus the graduate student learning outcomes are common to all the activities and are reported in Table 1.2 and were developed based on Baram-Tsabari and Lewenstein's six learning goals in science communication training: affective, content, methods, reflective, participatory, and identify goals.<sup>11</sup> In addition, graduate students were trained in awareness and communication with children and caregivers about food allergens, as many activities had an optional edible element.<sup>17</sup>

**Table 1.2:** Graduate student science communication learning objectives.

	Graduate students will be able to discuss why science communication
	is important, understand the challenges to science communication and
Affective Goal	be able to suggest ways to overcome those challenges. Students will
	also be motivated to continue participating in public engagement as a
	route for science communication.
	Graduate students will be able to effectively translate their ideas into
Content Goal	relatable and understandable terms and articulate them to the audience
	members.
	Graduate students will able to foster and encourage communication
Methods Goal	by exchanging ideas between themselves and the audience members
	through questions and conversation.
Reflective Goal	Graduate students will be able to articulate the importance and
	urgency of improving science communication on an academic level as
	well as on a societal level.
Participatory Goal	Graduate students will now actively seek to participate in other
	science communication opportunities and will use their
	communication skills to engage in meaningful dialogue with non-
	experts.
Identity Goal	Graduate students will now think of themselves as science
	communicators and will develop an identity of someone with the
	responsibility to properly and effectively communicate science.

For this program there were approximately 400 participants, however activity protocols are designed to be scaled up or down for varying participant needs. Importantly, participants are referred to in two groups: participants kindergarten-3<sup>rd</sup>

grade are considered "younger," while participants 4<sup>th</sup> grade-8<sup>th</sup> grade are considered "older." This distinction was intended to guide the activity instructors on how to better meet the intellectual needs and capabilities of varying age groups. Finally, the overall program was designed to be flexible so that this same event can be used again and adapted to changes in event space, resources, or audience demographics, including the ability to be brought as traveling exhibits to be implemented by resident program administrators or schoolteachers.

Each activity had a participatory element to keep young minds engaged, as well as a relatable context - something familiar to the general public that can facilitate understanding complex scientific concepts. The first two demonstrations, *Whip Up Your Proteins* and *Emulsions* introduce food chemistry topics by comparing what happens on an observational level to what is happening on a molecular level, while the *Vegetable Fermentation* and *Germs Can Glow* demonstrations introduces food microbiology in a simple and obtainable way, discussing topics sometimes not covered until a university level biology class. Finally, the *Milk Sensory* and *Orange Juice Processing* demonstrations reveal the influence of food process engineering on the organoleptic/sensory properties of beverages.

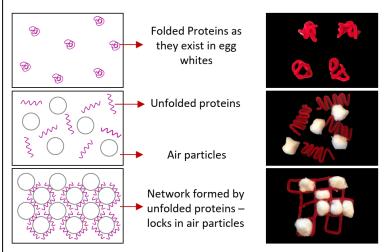
### 1.4 Activity 1 – Food Chemistry: Whip Up Your Proteins

**Materials and equipment:** pasteurized egg whites, graham crackers, marshmallow fluff, castor electronic eggbeater (needs outlet), three deep metal bowls, 2 oz. tasting cups, pipe-cleaners, and cotton balls. Optional: sugar, cream of tartar, cooked meringue or meringue cookies.

**STEM concepts:** polymer chemistry, proteins, colloids, dispersions

Learning Outcomes. Participants will understand what polymers are and establish the link between egg whites being proteins and proteins being polymers. Students will able to discuss protein structure in terms of the chemical interactions that cause proteins to fold and unfold and will be able to demonstrate how unfolded proteins are able to trap air to create a foam by using the interactive model provided. For older age groups, participants will be able to explain the components of a colloidal system and apply those concepts to other food systems. Participants will also be able to describe how additives like sugar and cream of tartar stabilize the colloidal food system.

Activity Background. Incorporating air into egg whites causes them to "fluff" up, but why? Egg whites are 10-15% proteins, whose structure is polymeric. A polymer is a macromolecule made of up repeating smaller molecules. These string-like proteins fold up into little clumps due to intermolecular forces within the chain, which is how they exist naturally in egg whites as seen in Figure 1.1, top. Beating egg whites



**Figure 1.1:** Protein folding and foam structure. Left: Schematic of air being incorporated into egg-whites to form a foam. Right: Analogous structure formed with cotton balls and pipe cleaners.

introduces air into the matrix which causes both the protein structure and composition of the system to change. The proteins denature, or unfold, and collect around the air bubbles. <sup>19</sup> Their now elongated structure allows them to entangle to form a temporary cross-linked network, sort of like a net. This network traps the air bubbles, giving rise to the foam and allowing the egg whites to remain fluffy for a while. The air bubbles in this system can be considered colloids, however their large size boarders the typical range of colloids. <sup>18</sup> Additives and processing conditions can stabilize this network, by promoting the retention of the air bubbles in the system. Examples of stabilizing additives include sugar (moisture control) or cream of tartar (pH control), and examples of processing conditions include application of heat.

**Methods.** To communicate these ideas to participants, two aspects must be considered. The first is demonstrating an *observational change*, in which participants *see* and *describe* the change from clear liquid egg whites to an opaque solid foam. The second aspect involves demonstrating what the participants cannot see, namely, the changes happening at a molecular level. For the first part of the demonstration, the instructor showed the participants the process of whipping egg whites. The instructor began by displaying liquid egg whites in a metal bowl and then using an electronic mixer to beat them until a stiff foam formed. Afterwards, the instructor set aside the bowl of egg whites to allow the foam to partially revert to a liquid. For older participants, ingredients such as cream of tartar or sugar can be added to different batches of egg whites before foaming to illustrate influence of formula composition on stability between these colloidal systems.

While the egg white foams rested, the instructor moved to the second part of the demonstration which exhibits the molecular changes in the egg-white matrix. The instructor constructed a pipe cleaner-cotton ball model ahead of time as shown in Figure 1.1, right. The pipe cleaners represent the polymer chains, and the cotton balls represent the air bubbles. The instructor began by displaying and passing around a folded pipe cleaner to resemble proteins as they exist naturally in liquid egg whites. Next, the instructor unfolded a pipe cleaner to illustrate the difference between the folded structure of a protein and the unfolded structure. Then, the instructor passed around a pre-constructed pipe cleaner crosslinked model holding cotton balls in the voids demonstrating how the proteins trap air bubbles which is the reason why egg whites can hold their foam for a while.

After this model-centered demonstration, the instructor guided the participants' attention back to the resting egg whites to show how some of the foam has turned back to liquid. This demonstration introduced the idea of *stability*. For older participants, the instructor compared the stability of plain egg white foam to the foams made with sugar and cream of tartar to illustrate how additives can stabilize colloidal systems.

**Discussion Points**. The instructor began this demonstration by explaining that egg whites are composed of 10-15% proteins, called albumins, which are polymeric in structure. During the demonstration, the instructor continuously encouraged participants to both ask questions and think critically about the change that is occurring. A strong emphasis was placed on the questions *what* is happening, *why* a change is occurring and *how* it occurs. For example, upon the beating of liquid egg whites, the instructor encouraged the participants to think about *what* is happening by asking "*What physical*"

changes do you notice as the egg whites are beaten? Is the color changing? How about texture and size?" It is important that the participants first grasped concepts of the observational changes that occur before the instructor moved on to the why and how explanations which covers the molecular changes that occur.

The instructor then addressed the question of why a change is occurring by first introducing other foods that share similar properties to beaten egg whites. For instance, the instructor asked the participants "what other types of foods can you think of that are fluffy?" After the participants were given time to think about similar food systems, the instructor explained other foams in foods such as whipped cream, meringues, marshmallows, and mousse. The instructor described a foam as air being incorporated and trapped in the egg whites. For older participants, the instructor went a bit farther and defined a colloidal system as a mixture with a dispersed substance, the colloid, uniformly spread out in another substance, the dispersion medium. Additionally, the instructor encouraged the older participants to name the colloid and dispersion medium in foams – the answer is air in a liquid or a solid medium. Beaten egg whites are a colloidal system of gas particles dispersed in a solid continuous phase, while whipped cream consists of gas particles dispersed in a liquid continuous phase.

Then the instructor communicated the *how*, leading with questions such as "what is changing on a molecular level when you whip the egg whites?" Using the pipe cleaner demonstration, the instructor communicated how the molecules in this system behave to make a change in the system. The instructor described that when air is being incorporated, the proteins unfold into an elongated form. These elongated protein strings get tangled which forms a temporary network that traps and holds the air bubbles. The

instructor asked questions to encourage the participants to critically think about why this effect might be happening for example "why are unfolded proteins more likely to tangle than the folded proteins?" The instructor also encouraged participants to pick up and play with the pipe cleaner and cotton ball props to promote a better understanding of the concepts. For older participants, the instructor followed up by explaining that additives like sugar, cream of tartar and heat stabilize the system. The instructor asked questions such as "what forces cause a protein to fold?" and "what about the sugar and cream of tartar prolong the re-folding of the proteins after beating?"

In addition to describing the concepts and ideas through the "what, why and how method," it was also important that the instructor continuously encouraged participants to describe what they have learned to other participants or to their parents. Doing so reinforces concepts and ensures that the participants have effectively learned something. At the conclusion of this activity, participants will be able to use their own language to describe complex ideas of food chemistry and polymer chemistry.

### 1.5 Activity 2 – Food Chemistry: Emulsions

**Materials and equipment:** oil soluble food coloring, water soluble food coloring, vegetable oil, deionized water, dishwashing liquid or detergent, powdered lecithin, egg yolks, effervescent heartburn relief tablets, and 12 clear 2-3 oz. plastic bottles.

**STEM concepts:** emulsions and emulsifiers, dispersions, hydrophobicity vs. hydrophilicity

Learning outcomes. Participants will be able to discuss the chemistry principles defining emulsions and will be able to demonstrate their knowledge by giving examples of emulsions in food systems. Participants will be able to apply food chemistry knowledge to understand what components of food ingredients provide functionality in food systems (e.g. lecithin in egg yolks). For younger age groups, participants will be able to explain that different food components are water-loving (hydrophilic) and others are water-hating (hydrophobic) and explain why those properties influence food systems. For older age groups, the participants will be able to explain the chemical interactions responsible for the immiscibility of molecules of different phases, such as oil and water. Older participants will also be able to describe the chemical interactions between emulsions and emulsifiers and apply that knowledge to food by describing how emulsifiers are utilized in common food systems.

Activity Background. Have you ever wondered why some salad dressings separate and need to be shaken before using them? The separation is natural, indeed, but what is causing it? Salad dressing is an example of a chemical phenomenon called an emulsion. An emulsion is any system where particles of one liquid phase are dispersed in a continuous medium of another liquid phase. Winaigrette style salad dressings are an "oil-in-water" emulsion, so when shaken, oil particles suspend themselves in the continuous aqueous (vinegar) phase. Separation in some vinaigrette style salad dressings happens because oil and vinegar are non-miscible, in other words oil is not soluble in vinegar. There also exist a class of molecules called emulsifiers which are compounds that have both water-loving (hydrophilic) and water-hating (hydrophobic) parts to them, and therefore stabilize an emulsion of two immiscible compounds from

separation. That is why vinaigrette-based salad dressings that have emulsifiers like egg yolk, lecithin, or other emulsifiers (e.g. xanthan gum) stay 'mixed'. Another example of this emulsifying effect is dish detergent which makes oil and water mix better and is the reason why you use it to help you clean the oily grease off your dishes! Like Activity 1, this demonstration also requires participants to draw a connection between an *observational change*, which is the immiscibility and separation of oil in water, and the *molecular* interactions.

Methods. The instructor prepared oil-in-water emulsions with and without emulsifiers (lecithin powder, egg yolk, dish detergent) to illustrate the observational repulsive interactions between oil and water and the effect of adding an emulsifier on emulsion stability. The instructor added a few drops of the oil soluble food coloring to the vegetable oil and filled the small plastic bottles half full of the colored oil and the other half with deionized water. The instructor capped off the bottles and demonstrated how to agitate the solutions to disperse colored oil particles in the continuous water phase. Participants were encouraged to participate by shaking the bottles themselves. The instructor asked the participants to place the bottles back on the table and observe the changes that occur while the oil and water phases separated. The instructor repeated this demonstration with the addition of emulsifiers (powdered lecithin, egg yolks, dish detergent). This demonstration was the foundation for the participants' introduction to emulsifiers.



**Figure 1.2:** Lava lamp demonstration. The blue oil phase and yellow aqueous (water) phase are immiscible, and thus naturally separate. The oil sinks to the bottom because it is denser than water, but when the sodium bicarbonate tablet is added, it sinks into the oil phase as it starts to dissolve. Gas bubbles which are less dense than both oil and water begin to rise and carry some of the blue oil with it creating a lava lamp effect.

The instructor led an additional "Lava Lamp" demonstration, especially suited for younger audiences (Figure 1.2). Using oil-soluble food coloring, the instructor colored vegetable oil and added it to a plastic bottle along with an equal amount of uncolored water. With the undivided attention of the participants (since the effect is short lived), the instructor added a sodium bicarbonate tablet to the plastic bottle, creating a lava lamp effect!

**Discussion Points**. The instructor began by asking participants: "Why don't oil and water mix? What can we do to make them mix?" and followed up by letting the participants try shaking the bottles of oil and water as hard as possible to get them to mix. Then, the instructor introduced the concept of emulsions and miscibility. Emulsions are a type of colloidal system, a mixture of (usually) two immiscible liquids,

one existing as small droplets dispersed in a continuous phase of another. The instructor explained *what* in this demonstration by first using the plastic bottle demonstration to illustrate the main concept, and then explaining that the components of an emulsion include two immiscible parts. The instructor further explained *what* behind the concepts of emulsions by asking the participants to "describe the changes that occur before agitation, during agitation, and after."

To illustrate the *why*, the instructor explained the immiscibility of oil and water, that the two phases in the plastic bottle separate because oil and water do not like to mix. For older age groups, the instructor defined the terms *solubility* and *miscibility* and asked participants to describe these terms in their own words to demonstrate their understanding. The instructor then described how the plastic bottle demonstration was an example of an oil-in-water emulsion, but there also exist water-in-oil emulsions, and even oil-in-water-in-oil emulsions and so forth. The instructor asked the participants to relate this same concept of emulsions to other food systems by asking questions such as "besides salad dressings, what other foods can you think of that might also be examples of emulsions?" The instructor followed up by describing the effect that takes place in salad dressing, expanded ideas of emulsions by describing how they can occur in other emulsion food systems for example in mayonnaise, milk, butter, and even hot dogs.

To get the older participants thinking about *how* these changes occur, the instructor shifted the focus to the behavior of this system on a molecular level. The instructor first asked the participants "what is the cause of repulsion between oil and water." Then, the instructor followed up by introducing concepts of polarity, hydrophobicity and hydrophilicity of certain molecules. Oil is a non-polar molecule

whereas water is polar, making their interactions repulsive in nature giving rise to emulsions.

Finally, the instructor introduced *emulsifiers*, explaining that they are molecules that have both polar and non-polar groups, enabling them to increase the favorability of the interaction of the oil and water interaction and therefore stabilize the emulsion. To illustrate this concept, the instructor used the same plastic bottle examples with oil in water and compared to similar bottles but with the addition of emulsifiers such as egg yolk, powdered lecithin, or dish detergent. Participants were asked to "describe the changes that occur before agitation, during agitation, and after", and compare these observations to the ones from the demonstration without any emulsifier included. The instructor then built on the why of "how does an egg yolk stabilize a mayonnaise emulsion" explaining that egg yolk contains lecithin, the emulsifying molecule responsible for stabilizing the emulsion. For older participants, the instructor asked the participants to build off their knowledge gained from the previous discussion about the repulsive interactions between hydrophobic and hydrophilic molecules to draw conclusions about the chemistry behind emulsifiers. At the conclusion of this activity, participants were able to describe the chemical and physical reasons for emulsions and emulsifiers.

### 1.6 Activity 3 – Food Microbiology: Fabulous Fermentations

**Materials and equipment:** non-iodized salt (e.g. kosher, sea salt), vegetables suitable for lacto-fermentation (e.g. cabbage, cauliflower, radish, carrot), non-chlorinated water (deionized or distilled), knife, cutting board, 64 oz. glass canning jars,

pickle weights (or an adequate substitute), vegetables mid-way through fermentation (actively bubbling), fermented vegetables, serving utensils, 2 oz. tasting cups, hand wipes, and hand sanitizer. Optional: fermented foods for display (e.g.: sourdough starter, SCOBY, 6 kombucha, cheese, yogurt, chocolate, coffee, bread).

**STEM concepts:** fermentation science, sensory science, microbiology, pH value,

Learning outcomes. At the completion of this activity and demonstration, participants will be able to discuss why food microbiology is an important branch of food science with implications in food preservation and food safety. Participants will be able to define the term fermentation and discuss the conditions under which relevant microorganisms can and can't grow, for example acidity (low pH) and salt (salinity) and describe how varying conditions can promote or inactivate microbial growth depending on the organism. Participants will be able to explain the principles involved in food preservation by fermentation, and how these preservation techniques also impart unique flavors to fermented foods. Participants will also be able to discuss how some microorganisms are pathogenic while others can be beneficial by promoting food safety or digestive health. Participants will be able to identify common grocery store items that are fermented such as yogurt, bread, pickles, chocolate, and coffee.

Activity Background. Many of the foods and beverages people consume have been fermented at some point between harvest and consumption, and this method of food preservation is not novel. In fact, there is evidence that food fermentations date back 12,000 years ago, a time before there was a scientific understanding of microorganisms and microbiology.<sup>20</sup> Indeed, fermentations play a role in food quality,

food safety and food preservation, and food microbiologists now have a better understanding of the microbiology principles behind food fermentations. Additionally, fermented foods present unique flavor profiles and are an exciting way to meld the worlds of microbiology, sensory science, and food preservation! The observational change can be described more like a sensory change since participants can use more than just their eyes to detect a change by smelling, feeling, and tasting the change in fresh vegetables before and after fermentation. It is important for the instructors of this exercise to understand the difference between the formal, biology definition of fermentation – an anaerobic cellular process in which an organic compound is broken down and energy is produced,<sup>21</sup> and the food preservation definition of fermentation – the microbial transformation of foods.<sup>20, 22</sup>

**Methods.** For this activity, the instructor prepared three sets of fermented vegetables: one approximately two weeks ahead of the event (fully fermented), one approximately 3 days ahead of the event (actively fermenting and bubbling), and one the day of the event (unfermented). Vegetables were washed, peeled (as needed), and cut into uniform sizes. A brine of 0.4 wt% non-iodized salt was prepared in deionized or distilled (not chlorinated) water. The cut-up vegetables were added to the 64 oz. mason jars and submerged in the brine. The pickle weights were placed on top of the vegetables to ensure full submersion. Jars were closed, but not sealed, and left to ferment at room temperature for the noted time (0, 3, or 14 days).

The fermentation jars were opened on the day of the demonstration and the fermented vegetables were served alongside of the fresh vegetables using 2 oz. tasting cups and serving utensils. One of each jar was left closed as a visual example. The

instructor had the participants taste the raw vegetable, followed by the 3 day ferment and ending on the 2-week ferment, while guiding the participants through the sensory differences of each type while introducing language such as "acidic" for the children to use to describe what they were tasting.

**Discussion points.** To first illustrate *what* changes occur due to fermentation, the instructor encouraged the participants to describe the sensory differences between the fermented carrots and radishes compared to the fresh carrots and radishes. For the older participants, the instructor asked them to think about not only *that* the taste and texture of the vegetable changes, but also *what* makes that change, *why* specific conditions are needed to make that change, and *how* that change occurs.

To introduce the concept of fermentation as it relates to food microbiology, food preservation, and sensory science, the instructor began by asking "what types of foods do you know of that are fermented?" The instructor then followed up by explaining the main concepts behind fermentation, emphasizing that not all microorganisms are bad, and that fermentation happens because of good microorganisms. In fact, food can be fermented by many different kinds of microorganisms, including bacteria (like pickles and yogurt), yeast (like bread or beer), mold (like miso or soy sauce), or a mix, sometimes called a SCOBY (symbiotic culture of bacteria and yeast). The instructor then focused the participants attention to thinking just about fermented vegetables, which are preserved by a kind of bacteria called lactic acid bacteria, which (unlike some the microorganisms that might make us sick or spoil our food) thrive in a salty and sour (acidic, low pH) environment of a pickle brine. For older participants, the instructor went a step further to ask the students about the three jars of fermenting vegetables using

questions such as "why are there bubbles in the 3-day ferment? What are some differences in how the vegetables and brine look and taste in the fresh, 3-day, and 14-day ferments?" The instructor then explained the why by describing the chemical processes of lactic acid bacteria breaking down the sugars in vegetables to produce lactic acid and carbon dioxide. As fermentation time increases the amount of lactic acid bacteria increases and as a result acidity increases, the sugar in the vegetables decreases and so does the amount of carbon dioxide produced.<sup>23</sup>

The instructor then went on to introduce, to participants of all age groups, other foods that are fermented that one might not expect such as coffee, yogurt, bread, chocolate and cheese! It was explained that all fermentations do not follow the same process, and not all fermentations use a salt brine. The instructor distinguished between different types of fermentation and highlighted that this demonstration was an example of *wild* lactic acid fermentation, in which the lactic acid bacteria that transformed the vegetables into pickles was naturally present on the vegetables. The instructor then explained that wild fermentations differ from other types of fermentations where cultures of specific microorganisms are added to raw materials to initiate the fermentation, which is the case for most commercially produced fermentations that you can buy in the grocery store.

The instructor then introduced the concept of food preservation and food safety

– how food preservation techniques are used not only to change the taste of foods, but
also to prevent the 'bad' microorganisms from making our food unsafe to eat. The
instructor then explained that in vegetable fermentations, the vegetables are preserved
by lactic acid bacteria, which not only changes their flavor and texture but can also

protect them from growth of spoilage or pathogenic microorganisms. For the older participants, the instructor encouraged them to think about how microbes promote environmental changes during fermentations which in turn can protect the food from spoilage to illustrate how fermentations are used to preserve foods. At the conclusion of this activity, participants will be able to articulate the difference between a wild-fermentation and a cultured fermentation and will be have gained the proper language to discuss the sensory differences between raw and fermented foods. Participants will also be able to explain how fermentation science is a branch of microbiology. Participants will be able to articulate that the growth of non-pathogenic bacteria produced by fermentations prevent the growth of harmful and spoilage microorganisms and will be able to explain that the conditions for this in vegetable fermentations require a salty environment.

### 1.7 Activity 4 – Food Microbiology: Germs Can Glow? And Where Did They Go?

Materials and equipment: Glo Germ<sup>™</sup> (DM Internationally), UV flashlights, hand wipes, paper towels, hand sanitizer, Stop Germs! Wash Your Hands!© Handout Centers for Disease Control and,<sup>24</sup> Story of Your Dinner Food Safety Tips Handout© Partnership for Food Safety.<sup>25</sup> Access to a sink with soap.

**STEM concepts:** microbiology, food safety, public health

Learning outcomes. After this interactive demonstration, participants of all age groups will be able to discuss basic principles in food safety and public health. Participants will be able to describe routes of contamination, discuss methods for controlling hazards, evaluate safe conditions and articulate the reasons why

handwashing is more effective than using only hand sanitizer. Participants will also be able to explain how cross-contamination can occur from someone with dirty hands to another person or the food they touch.

**Activity Background.** Washing your hands for 20 full seconds is important to keep from getting sick, but why? Handwashing is the best way to remove harmful microorganisms from your hands, but only if done thoroughly and properly. It is important to pay special attention to the more difficult areas to clean, for example between fingers and around the fingernails. Glo Germ<sup>TM</sup> (DM International), a lotion containing small non-toxic particles (1-5 mm, approximately the size of bacteria) that glow when exposed to UV-light, is a useful interactive tool to demonstrate the importance of washing your hands thoroughly. Once applied, the lotion is not visible under normal light making it impossible to see if it has been completely removed by handwashing or sanitizing. This concept is the same for the microbes on your hands, it is impossible to simply see whether your hands are completely clean. Using Glo Germ<sup>TM</sup> (DM International) and a UV-light, the difference between proper hand washing, improper hand washing, and use of hand sanitizer in reducing the transfer of germs either by skin-to-skin or skin-to-surface contact can be demonstrated. The observational aspect is clear in this case, it is the UV light applied to hands before and after hand washing, skin-to-skin contact or skin-to-surface contact that illustrates how germs can be spread.

**Methods.** This activity consisted of two demonstrations, the first highlighted how handwashing with soap and warm water is more effective than hand sanitizer. The instructor had the participants apply the Glo Germ<sup>TM</sup> (DM International) to their hands

thoroughly. Then, the instructor shined the UV-light on their hands so the participants could see the streaks left by the Glo Germ<sup>TM</sup> (DM International). Some of the participants washed their hands at a sink using soap and warm water for 20 full seconds, while other participants were instructed to try to remove the Glo Germ<sup>TM</sup> using paper towels, wipes, or just hand sanitizer. After both handwashing protocols, the instructor shined the light on the participants' hands again to illustrate how much more effective washing with soap and warm water is than hand sanitizing or wipes alone.

Glo Germ<sup>TM</sup> (DM International) can also be used to show how microbes can be transferred through human contact, for example a handshake, or spread on surfaces as illustrated by the second demonstration. The instructor asked some participants to apply Glo Germ<sup>TM</sup> (DM International) and proceeded to shine the UV-light to show the streaks left by the gel. Fist, the student who had applied the gel was instructed to shake the hand of a student without the gel. The instructor then shined the UV-light on the student with the previously clean hand to show that the gel had transferred from the original student to the student with the clean hands. Next, the instructor shined the UV-light on a clean surface and instructed a student with the gel to touch the previously clean surface. Then the instructor shined the UV-light on the surface to show how microbes can be spread through contact with commonly touched objects.

**Discussion Points**. Before using the gel, the instructor began by asking participants why they think it is important to wash hands thoroughly. Questions such as "what can happen if you do not wash your hands," "for how long should you wash your hands" and "is hand sanitizer or wiping with a wipe or paper towel as effective as hand washing" were good to have participants start thinking through concepts themselves.

The instructor emphasized that one of the most important reasons to wash hands is to avoid the spread microorganisms that can get us sick, since one way these pathogens are spread is through contact. The instructor ensured the participants understood that foodborne pathogens can lead to illness including the usual symptoms of diarrhea, vomiting, nausea and sometimes more serious outcomes.

The instructor then went on to explain the proper way to clean hands. Washing with soap and water for 20 seconds is the gold standard for hand washing, with hand sanitizers as an alternative only when running water and soap are not available. While hand sanitizers can inactivate many microorganisms, and are quick and easy to use, they cannot (unlike hand soap) remove grease and dirt that makes microorganisms stick on hands and other surfaces. Using the handwashing guide developed by the CDC (Centers for Disease Control), the instructor explained to the participants the recommended way to wash hands in five steps: wet, lather, scrub, rinse and dry. The washing step should go on for about 20 seconds, or for the duration of singing "Happy Birthday" twice.<sup>24</sup>

Before the handwashing versus hand sanitizing/wiping demonstration, the instructor explained to the participants that Glo Germ<sup>TM</sup> (DM International) is a simulation for bacteria (and not actually bacteria) and is intended to illustrate the effectiveness of different methods of handwashing as well as how microorganisms can spread by contact. The gel contains a dye that glows under UV light and acts like dirt and grease that gets trapped on your hands that you cannot see.<sup>26</sup> The above points were reinforced before and after each demonstration. Afterwards, it was clear to the participants that hand washing was the much more effective way at removing Glo Germ<sup>TM</sup> (DM International) compared to hand sanitizing.

The instructor emphasized that hand sanitizer is a good option if there is no sink nearby, however it should not replace hand washing. The instructor explained *why* by telling the participants how washing hands can remove dirt and grease from hands that tends to trap bacteria. Additionally, the instructor used both demonstrations to explain that although Glo Germ<sup>TM</sup> (DM International) does not show actual bacteria on hands, it acts a simulation and provides a way for scientists and participants to learn how long to wash and how thorough to be. At the conclusion of this activity, participants were able to articulate the scientific reasons why handwashing promotes good public health and is a good food safety practice. Participants could also discuss how bad microorganisms can transfer from person to person, from person to foods, and from person to surfaces like school desks.

#### 1.8 Activity 5 – Food Processing and Sensory Science: Milk Sensory

**Materials and equipment:** 2% milk and 2% Lactose Free Milk (of the same brand), 2% shelf stable milk or 2% Ultra-High-Temperature (UHT) milk (optional), ice, 2 oz. tasting cups, and a cooler.

**STEM concepts:** enzymes, food chemistry, sensory science, food processing **Learning Outcomes.** After participating in this activity, participants will be able to discuss the variability in the sensory experience of different milks using explanations involving food processing techniques. Participants will be able to discuss how different sugars are perceived to be sweeter or less sweet than others and relate that back to the sweeter flavor in lactose-free milk. Participants will be able to describe the

difference in steps (unit operations) used to produce lactose free milk compared to 'regular' milk.

**Activity Background.** From "Milk, it does a body good" to the "Got Milk?" campaigns, <sup>27,28</sup> milk is a healthy drink for many kids, high in protein, low in sugar, and full of many important vitamins and minerals. However, 65% of the human population suffer lactose intolerance<sup>29</sup> which is when the sugar lactose, naturally found in milk and other dairy products, can make them feel sick. To make it possible for people with lactose intolerance to drink milk, the dairy industry found a food processing technique using the enzyme lactase to break down the lactose into glucose and galactose.<sup>18</sup> Glucose and galactose are sugars that are more easily digestible even for people with lactose intolerance, <sup>30</sup> and thus allows lactose intolerant consumers to enjoy dairy milk. In fact, lactase is present in the body so that people can digest the lactose in milk. People who are lactose intolerant have less lactase and thus cannot breakdown dairy products as easily. When you use the lactase enzyme to make lactose-free milk, the conversion of lactose to glucose and galactose also changes the perceived flavor since different sugars taste sweeter than others, for example glucose and galactose taste sweeter than lactose.<sup>31</sup> These flavor changes are not necessarily a bad thing. Many consumers prefer the taste of lactose-free milk because it is sweeter. Other processing techniques can also cause the flavor to change. After the lactase enzyme has done its work, lactose-free milk is treated to a second heat treatment to inactivate it (all pasteurized milk is heated to inactivate spoilage and pathogenic microorganisms, lactose-free milk gets a second heat treatment). When milk is heated to a certain temperature, the whey proteins are broken down via disulfide bond breakage which, in turn, can causes an eggy-caramel flavor,

the egginess coming from the disulfide bond breakage and the caramel flavor coming from the heating of the sugars.<sup>18</sup>

**Methods.** This sensory demonstration outlined the differences between the milk samples processed using standard pasteurization and those processed using pasteurization, the enzyme treatment, and a second heat treatment. The instructor prepped the tastings ahead of time by pouring each of the milk varieties into the 2 oz. tasting cups, ensuring that all milk samples were to be tasted at the same temperature. The cups were organized on the table so that the instructor knew which sample was which, but the participants did not. The original containers with the labels were also placed on the table so that the participants knew what they were tasting, even if they were not aware of the order in which they were tasting them.

For the tasting, the instructor asked participants to begin tasting the milks, starting with the 2% milks. Participants were asked to taste and compare the flavors and aromas of the milks. The instructor engaged the participants using the discussion points below.

**Discussion points**. During the white milk tasting, the participants were asked a series of sensory questions to get them to understand *what* is the difference between the milks including "which of the milks tastes sweeter?" "Which milk do you think contains more sugar?" And "do any of the milks have an eggy taste?" After the students were able to articulate the sensory differences between the milks, the instructor followed up by explained why there exist different types of milk. Lactose free milk is provided so that customers who cannot digest lactose may enjoy milk, whereas shelf-stable milk is good for situations when refrigeration is difficult. The instructor then moves on to

describe *how* each milk is processed differently. In lactose-free milk, lactase enzyme is added which converts the lactose to glucose and galactose which taste sweeter than lactose. In the UHT milk, part of the processing involves heating the milk which can denature the proteins. Proteins exist in milk as folded structures due to intermolecular disulfide bonds, meaning a sulfur-sulfur bond. When the disulfide bond is broken is and replaced with a hydrogen, the proteins unfold. The generation of a sulfhydryl group, or sulfur-hydrogen group, is what causes the cause an eggy flavor. The instructor emphasized the point that different processing techniques can greatly change the sensory perception of a food product. Then, the participants were asked to use their new knowledge to match the milk tasting to the product label.

To further explain Lactose, the instructor explained what lactose is first by simply asking the participants "what is lactose?" and followed up with questions like "where is lactose most commonly found?" "What do you think lactose taste like?" The instructor then moved on to why people have difficulty digesting lactose by introducing the word enzyme and describing how one common enzymatic function was breaking down bigger molecules into smaller ones. This process is how the sugar lactose is broken down to the sugars glucose and galactose. A strong emphasis was put on the fact that enzymes can be used in food processing, just like heating milk in pasteurization. The participants were then asked to guess why breaking down sugars into new sugars might change the perceived sweetness and were introduced to the idea that different sugars have different sweetness intensity. The instructor also introduced the interesting fact that taste perception is a result of many things, including age, dietary habits, and even genetic differences between people. Taste perception occurs when "taste

molecules" interact with receptors on tongues, otherwise known as taste buds, and the structures of these receptors can differ from person to person based on genetics. <sup>18</sup> Thus, perceived sweetness can vary not only due to the compounds present in the milk but can also vary from one individual to another.

At the conclusion of this activity, participants were able to explain why different milk options (lactose free, shelf stable) were available for consumers and that they required different processing techniques. Additionally, the participants will be able to describe how those different processing techniques can change the chemistry of the sugars in the milk which then affects the taste of milk.

# 1.9 Activity 6 – Food Processing and Sensory Science: Life's a Squeeze.

**Materials and equipment:** orange juice processed and packaged in at least 3 different formats\* (see Table 1.3), 2 oz. tasting cups, pitchers for waste juice. \*It is helpful to keep the brand of orange juice consistent.

**STEM concepts:** food processing, sensory science, process engineering/unit operations, food safety, microbiology, food chemistry, and materials science.

**Table 1.3:** Types of orange juice by processing, packaging, and storage conditions.

Orange Juice Type	<b>Processing Unit Operations</b>	Packaging	Relative Shelf Life
Fresh Squeezed	1. Juice Orange	Varies	Shortest
Ready to drink	2. Cold Fill → Bottle		
Refrigerated			
Cold Pressed Ready	1. Juice Orange	Plastic Bottle	
to Drink	2. Cold Fill → Bottle		
Refrigerated	<ol><li>High Pressure Processing</li></ol>		
	1. Juice	Plastic Bottle	
Ready to Drink	2. High Temp Short Time		
Refrigerated	pasteurization (e.g. 165°F/15s)		
	3. Cold Fill → Bottles		
	1. Juice	Plastic Bottle	
Ready to Drink	2. Pasteurization	(that can	
Shelf-Stable	(e.g. 180° F, 3 min)	withstand higher	
	3. Hot Fill → Bottle	temperatures)	
Frozen Concentrate	1. Juice	Paperboard with	
	2. Evaporation	double seam	Longest
	3. Fill	metal ends	
	4. Freeze		

Learning outcomes. After this activity, participants will be able to discuss and relate principals of microbiology, process engineering, packaging technology, and sensory science. Participants will be able to discuss how food processing control for pathogens and how different processing techniques result in products with varying shelf-lives, packaging requirements, and storage conditions. Participants will be able to list various packaging methods for beverages, specifically orange juice, and describe the reasons why products require different packaging as they relate to food processing, quality, and safety. Additionally, participants will be able to explain how different processing techniques cause sensory changes using principles of food chemistry and sensory science.

**Activity Background.** Orange juice is not only delicious but a healthy source of Vitamin C. However, vitamin C is light and oxygen sensitive and thus requires

packaging with a high oxygen and light barrier. Common materials used for orange juice packaging include aseptic boxes, gable-top coated paperboard cartons, retort pouches, aluminum cans, glass bottles and plastic jugs.<sup>33</sup> This activity introduces both refrigerated and shelf stable orange juice packaged in plastic bottles (semi-rigid polymer) as well as paperboard with double seam metal ends for frozen orange juice concentrate (see Table 1.3).

It is interesting that the same brand orange juice in different packaging tastes slightly different. It is important that the foods we consume are safe to eat, thus different processing techniques are selected for the shelf-life or storage you want, for example shelf-stable, refrigerated, or frozen and this in turn leads to package choice. Freshly squeezed orange juice that is simply squeezed and placed in a bottle for sale has a much shorter shelf life than juice that has undergone pasteurization at 180° F for 3 min and placed in the bottle while still hot (a process called hot-fill). This processing step controls for pathogens in the juice which is why the product can last without refrigeration much longer than freshly squeezed. Since the juice is poured in when still hot, the bottle much be made of a polymer (scientific term for the more colloquially used "plastic") that will not soften or melt upon contact. Other times, orange juice is processed by either high pressure processing (subjecting orange juice to high amounts of pressure) or high temperature short time pasteurization (165°F for 15 seconds) to control for microbes. These products are filled in the bottle cold (a process called coldfill) and can stay safe to drink longer than freshly squeezed orange juice but still require refrigeration.

Although these processing techniques make orange juice safe to consume and last longer on the shelves, heating processes can change other elements in orange juice and can result in a loss of key flavor compounds such as limonene into the packaging, loss of vitamin C from oxygen which commonly happens in plastic bottles, or a loss of bright orange color which can turn to a dull brown-orange color, a process called non-enzymatic browning. These changes in turn have an effect on the sensory characteristics of orange juice and thus, processing and packaging choices can alter the way it tastes!

**Methods.** The instructor prepared the tasting cups before the event began. Ensuring that each type orange juice is the same temperature, the instructor lined up rows of each type of orange juice in front of their respective food packages. Once participants started arriving at the table, the instructor simply guided each student through the orange juice varieties starting with the color of each juice followed by the aroma and then flavor.

**Discussion points**. To first get the students to understand *what* the sensory differences were between the orange juice samples, the instructor guided the participants through some key elements of sensory science. The instructor began by asking the participants to describe any visual differences between the juices. Next, the instructor asked the participants to taste the juice and to pay close attention to changes in the orange flavor, acidity, sweetness of each juice. After the participants grasped *what* differences could be detected, the instructor encouraged the participants to think about *why* companies might want to package the same product differently. After the participants began thinking about different packaging needs, the instructor explained that the first and foremost goal of packaging is to prevent microbial contamination so

that the product is safe to consume, while packaging also keeps the product fresh and delicious.<sup>34</sup> The instructor then explained that orange juice is high in Vitamin C which is good for our bodies but also reacts with oxygen. This reaction causes browning as well as changes in flavor and aroma. The instructor then went on to explain that orange juice packaging can be used to prevent Vitamin C degradation and packaging (and thus processing) can be altered to extend the shelf life of orange juice.

After describing packaging needs, the instructor explained how different orange juice is packaged to accommodate shelf life requirements. To engage the participants, the instructor began by asking the students "which orange juice packaging do you think is needed for the longest shelf life? How about for refrigerated juices?" Next the instructor explained that for freshly squeezed orange juice, the orange is juiced and then poured into a container, which may vary in type as the shelf life will be short. Then the instructor made the contrast with more processed orange juice options by starting with other refrigerated examples. Plastic refrigerated orange juice bottles are filled and then subjected to high pressure processing, a food safety technology that uses 5,000-6,000 bars of pressure to inactivate microbial cells. However, this amount of pressure does not kill bacterial spores and thus refrigeration is needed to keep this product safe.<sup>34</sup> The shelf life of refrigerated orange juice can be extended even more if it is pasteurized, meaning heated to a specific temperature for a specific amount of time (165°F for 15 seconds). For shelf stable orange juice, higher temperatures and often longer times are used.

Next, the instructor asked the participants to speculate *how* differences in processing might affect flavor. The instructor reminded the participants that degradation

of Vitamin C can cause sensory changes in orange juice. Finally, the instructor introduced concepts of sensory science to the participants by first asking them questions such as "why is it important to talk about the sensory experience of food products?" For older age groups, the instructor asked for a description of specific sensory experiences that might be expected from a product such as orange juice. The instructor used ideas developed from this discussion to tie food processing and sensory experience together while integrating concepts of materials science and microbiology. A strong emphasis was put on how these and other branches of food science are intertwined and co-dependent. The instructor also encouraged participants to consider how consumer expectations might influence processing techniques or product packaging.

At the conclusion of this activity, the participants will be able to articulate how sensory science relates to processing techniques which vary based on food safety and shelf-life requirements. Regarding food packaging and processing, participants will be able to explain the different properties of each type of processing that each beverage package requires. Additionally, participants will be able to explain why sensory experience is important in foods.

#### 1.10 Conclusion and Future Directions

The primary outcome of this six-activity event was to introduce topics in food chemistry, food microbiology, and food process engineering & sensory science to the non-expert public, including K-8<sup>th</sup> grade participants and their adult caregivers in an informal learning environment. In parallel, and a major goal of this event, was that by acting as science communicators for the activities, food science graduate participants

had the opportunity to grow their science communication skills by explaining complex topics in both fundamental (e.g. physics, chemistry) and applied (e.g. food engineering, fermentation science) STEM fields through the more contextual lens of food science. This preliminary event was held at the Ithaca Sciencenter, as one of our goals was to reach a wide age-range audience and it has been shown that people of all ages can effectively learn science in informal environments, such as a science museum.<sup>7</sup>

Learning objectives for both the participants (the science learners) and the graduate students (the science communicators) were set prior to both conducting the activity and in the process of designing the activities. In fact, part of the graduate students' training in science communication was designing and writing learning objectives for their activities since doing establishes a framework for both communication and assessment. Further, it was not only important for there to be communication, but also engagement between the graduate students and the audience members, meaning there was a dialogue between them. It was toward the goal of effective engagement that motivated the suggested discussion points in the activity protocols since learned materials are reinforced when the learners must engage in conversation and repeat back in their own words the knowledge they have gained. In observation of the Sciencenter event, participants were excited to ask questions about the demonstration and participate with hands-on activities while graduate students were organized and enthusiastic with their answers. The adult caretakers also showed engagement during the demonstrations by not only encouraging children to repeat back what they have learned, but also asking questions related to their own daily life experiences. For example, at the Whip Up Your Proteins demonstration, one adult asked

why they add cream of tartar to stabilize their whipped egg whites. This example is indicative of the adaptability of these demonstrations and again of the importance of presenting science using relatable topics. The importance of adult engagement should not be overlooked. One of the goals of this program was to teach and inspire young minds to question their surrounding world. If students get into the habit of asking questions from a young age, their scientific potential to ask meaningful questions as they grow and learn only greatens. Students can be encouraged to be inquisitive in the classroom, but ultimately it is a skill that must be nurtured and encouraged at home as well. And thus, if educators can also encourage caregivers to ask insightful questions, setting an example for young students, that is only further reinforcement of these types of educational practices.

Another important goal for this program was that it had long-term impacts which is the reason each activity protocol was designed to be adaptable for different event circumstances. Providing options in the activity discussion points to deepen the material for more advanced learners, or to keep it simple yet informative for younger participants with a less extensive scientific background is one example of the versatility of this program. Additionally, each protocol provides flexibility in quantities to scale appropriately for the volume and space of future events or to adjust for different budgets to further promote science learning in informal environments. It is also important for science engagement to be accessible and easily implemented, not only by experts but also by other educators trying to improve their science communication skills. It is widely accepted that as people become more comfortable and literate with scientific topics, they develop a greater appreciation for the sciences and will understand and appreciate

the important role of science in bettering our world<sup>6</sup>. Thus, it is imperative that we develop programs to familiarize as many people as possible to scientific topics<sup>35</sup> as well as create opportunities to equip science educators to better science communicate. These organized activity protocols serve not only as guidelines for events that may be hosted in science museums, but also may function as kits that can be assembled and sent to resource-limited schools and programs. As a result, program administrators or schoolteachers can become equipped to teach students about food science, an area that is less common in elementary curriculum. Thus, not only does this program promote the science communication skills of graduate students and public engagement in science but can allow educators to strengthen their science communication skills and can widen the science topics young children are exposed to. In conclusion, the event achieved engagement between both graduate student instructors and participants of all ages suggesting effective science communication was practiced. Training experts to effectively communicate their knowledge is crucial not only for professional development, but also for scientific advancement and to achieve a better-informed public.

#### Acknowledgements

This work was funded in part by the United States Department of Agriculture National Institute of Food and Agriculture under award # 2019-38420-28975, and in part by the Department of Food Science at Cornell University. The original program was hosted at *The Sciencenter* in Ithaca, NY with special help from Coordinator, Lyla White and Executive Director Michelle Kortenaar. The graduate student volunteers

from Cornell's Graduate Food Science Program were Rachel Carson, Catherine Dadmun, Devin Daeschel, Margaux Ehrlich, Brenna Flynn, Ian Kay, Sarah Kozak-Weaver, Timothy Lott, Meghan McGillin, Mariely Medina, Ann Van Le Nguyen, David Parker, Jessica Rafson, Elizabet Reyes, Autumn Rudlong, Jonathan Sogin, Zhixin Wang, Zirui Ray Xiong, and Mohammad Yaghoobi. We also thank Dr. Bruce V. Lewenstein of the Department of Communication of Cornell University for sharing his expertise on science communication.

#### **Author Contributions**

A. J. Macbeth designed the program and each activity protocol and led the training of graduate student science communicators. H. S. Zurier provided substantial critical revisions for this work, especially to ensure the science in each activity protocol is accurate. E. Atkins contributed to the design of this program by offering her expertise in childhood education. S. R. Nugen contributed substantially to the revising of this work. J. M. Goddard (corresponding author) provided substantial support of the design, drafting and revision of this work as well as the final approval of the version to be published.

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#### CHAPTER 2

# GENERAL METHOD FOR EMULSION POLYMERIZATION TO YIELD FUNCTIONAL TERPOLYMERS<sup>1</sup>

#### 2.1 Abstract

Copolymerization methods are used to impart specific, desired functional properties (e.g. mechanical or bioactive) to a material for targeted applications in biomedicine, food and agriculture, consumer products, advanced manufacturing, and more. Many polymerization methods exist to achieve tailored copolymer architectures. Of them, emulsion polymerization offers unique and industrially convenient features that make for easily scalable processes because the synthesis occurs in water and the latexes usually do not need further purification. Because of the breadth of copolymer architectures and thus wide range of potential applications for latexes produced by emulsion polymerization, there is great value in defining general methods to permit consistency and optimization of these processes. Herein we present a general emulsion polymerization method for synthesis of a copolymer consisting of three functional monomers, suitable for adaptation to alternate base chemistries, curing chemistries, and functional ligands.

• Our synthesized copolymer includes a glycidyl methacrylate (GMA) monomer functionalized with a metal-chelating iminodiacetic acid (IDA) ligand, a UV-curable

<sup>&</sup>lt;sup>1</sup> **Macbeth, A. J.,** Lin, Z., Goddard, J. M. (2020). *General Method for Emulsion Polymerization to Yield Functional Terpolymers*. \*Submitted for publication.

- monomer, 4-benzoylphenyl methacrylate (BPM), and an inert hydrophobic monomer, *n*-butyl acrylate (BA).
- The presented synthesis route demonstrates a general polymerization method that can be modified to copolymerize alternative functional monomers to create multi-functional polymers.

# 2.2 Specifications

**Table 2.1:** Method Details

Subject Area	ChemistryMaterials Science, Agricultural and Biological Sciences	
More specific subject area	Polymer Chemistry	
Method name	d name General Emulsion Polymerization of Functional Copolymers	
Name and reference of original method	Lin, Z., & Goddard, J. M. (2018). Photocurable coatings prepared by emulsion polymerization present chelating properties. <i>Colloids and Surfaces B: Biointerfaces</i> , 172, 143-151.	
Resource availability	Mechanical Stirrer: available through ChemGlass  Reactor Stand: <a href="https://chemglass.com/chemrxnhub-support-stands-benchtop-reactors?AspxAutoDetectCookieSupport=1">https://chemglass.com/chemrxnhub-support-stands-benchtop-reactors?AspxAutoDetectCookieSupport=1</a> Glass stir rod: <a href="https://chemglass.com/stirrer-shafts-polished-10mm-chem-stir?sku=CG-2078-05">https://chemglass.com/stirrer-shafts-polished-10mm-chem-stir?sku=CG-2078-05</a> Agitator: <a href="https://chemglass.com/agitator-ptfe-anchor-style">https://chemglass.com/agitator-ptfe-anchor-style</a> 2-part, 5-neck, 300mL round bottom flask was ordered custom through ChemGlass.  Personal Protective Equipment: Long pants, lab coat, closed toed shoes, eye protection (safety glasses) and gloves. Gloves can be natural rubber, butyl rubber, nitrile rubber, neoprene or polyvinyl chloride (PVC).  Reagents: Purchased from SigmaAldrich and Fisher Scientific and used as received.	

# 2.3 Background and Additional Information

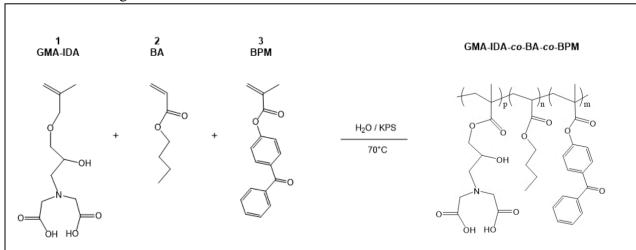
Copolymers enable combining the functional or mechanical properties of more than one homopolymer in a single material making them useful in a wide array of

applications that require multiple polymer characteristics such as clothing, rubbers, latex paints, materials for automobiles, electronics, furniture, construction materials, packaging, adhesives and biomedical applications.<sup>2,3</sup> For example, some polymer functionalities useful for applications in active packaging could include a metal chelating ligand that improves the food's shelf-stability and a UV-curable moiety that promotes its application in high-throughput industrial processes because they do not require solvents nor high energy inputs to cure.<sup>4-6</sup>

There are many polymerization methods employed to achieve copolymer architectures, however emulsion polymerizations are advantageous because their resulting latexes do not generally need further purification making this a viable option for high-throughput processes and synthesis occurs in water, and thus is a more environmentally friendly process. Emulsion polymerizations proceed via radical polymerization and are widely used to synthesize industrial polymers for examples adhesives, paints, binders, textiles and construction materials. Although there are strong advantages to emulsion polymerization, these syntheses generally involve complex reactor set-ups that require many steps and which can lead synthesis inconsistency. Thus, standard methods for copolymer syntheses via emulsion polymerization are needed to improve the consistency and optimization of these processes.

Herein we present a general emulsion polymerization method to synthesize a copolymer consisting of three functional monomers that can be adapted based on the needs of the film. Our copolymer includes a glycidyl methacrylate (GMA) monomer functionalized with a metal-chelating iminodiacetic acid (IDA) ligand, *n*-butyl acrylate

(BA), and a UV-curable monomer, 4-benzoylphenyl methacrylate (BPM) as shown in Figure 2.1. Although the presented copolymer shows specific functionalities intended for a specific application in metal chelating, antioxidant active packaging coatings, this method is intended to provide a general emulsion copolymerization process with tips and notes. With slight modifications, the presented methods may be employed to synthesize other functional copolymers via emulsion polymerization. For example, bio-based monomers, or functionalities such as thermocuring, antimicrobial or non-fouling ligands, or even biocompatible monomers. Functional copolymers have an extensive range of possible applications some of which include food packaging, food safety, water treatment, biomedical implants, or functional polymer coatings. Providing standardized methods lend detailed information on reactor set-ups and method validation that can serve as a foundation for further functional copolymerization syntheses with applications in human health, defense, food and agriculture, and advanced manufacturing.



**Figure 2.1:** Copolymerization of poly(2-propenoic acid,2-methyl-,3-[bis-(carboxymethyl) amino]-2-hydroxypropyl ester-*co-n*-butyl acrylate-*co*-4-benzoylphenyl methacrylate) (GMA-IDA-*co*-BA-*co*-BPM) via emulsion polymerization initiated by potassium persulfate (KPS).

#### 2.4 Part I

#### Synthesis of Monomer 1: Glycidyl Methacrylate – Iminodiacetic Acid (GMA-IDA)

<u>Reaction Duration</u> – 3 hours total: 0.5-hour synthesis preparation + 1-hour synthesis + 1.5-hour purification

**Figure 2.2:** Mechanism for the synthesis of monomer 1: glycidyl methacrylate-iminidiacetic acid (GMA-IDA) beginning with a neutralization of iminodiacetic acid (left) followed by an epoxy ring opening to join GMA and IDA together (right).

#### Reagents

Sodium hydroxide pellets, deionized (DI) water, iminodiacetic Acid (IDA) (98+%), glycidyl methacrylate (GMA) (97%), concentrated hydrochloric acid (trace metal grade), and acetone.

<u>NOTE:</u> this general method can be adapted to synthesize other functional (e.g. antimicrobial, biotinylated) monomers using the glycidyl methacrylate base monomer.

<u>NOTE:</u> many hazardous reagents are used in this method; carefully review and post SDS (safety data sheets) for the safety of the individual performing the synthesis and others in the laboratory.

#### Equipment

The reactor consists of a 300 mL two-part, 5-neck round bottom flask, overhead mechanical stirrer, glass stirring rod and anchor style agitator, reactor stand, hot plate, battery operated thermometer with clip, oil bath set to 65°C, condenser, addition funnel, 3 rubber septum stoppers. Other equipment includes 250 mL beaker, magnetic stir bar and stir plate, parafilm, 10mL mechanical pipette, 5mL mechanical pipette, ring stand with ring attachment compatible with 500mL separatory funnel, 500mL separatory funnel, clean Erlenmeyer flasks for extraction, Buchner funnel, filter paper, and a vacuum line.

<u>NOTE:</u> the nitrogen line is not necessary for Part I, however it will be utilized later in Parts II and III.

#### **Preparation**

1. Assemble the reactor as shown in Figure 2.3, but without the nitrogen line. Plug the three, free necks with rubber septum stoppers. Heat the oil bath to 65°C.

<u>NOTE:</u> the reactor is set up in a dedicated fume hood, and should only be performed by someone trained to safely conduct the synthesis.

- 2. Prepare a 50mL solution of 2M NaOH:
  - In a 250mL beaker equipped with a magnetic stir bar, add 4g of NaOH pellets to 50mL DI water.

ii. Cover the beaker with Parafilm® and place on a magnetic stir plate set to 350 rpm. Let stir until all the NaOH is dissolved, about 10 minutes. <u>TIP #1</u>: Mixing NaOH into water is highly exothermic and will produce heat.

<u>TIP #2</u>: It is recommended to perform this step in a secondary containment vessel in case of spills.

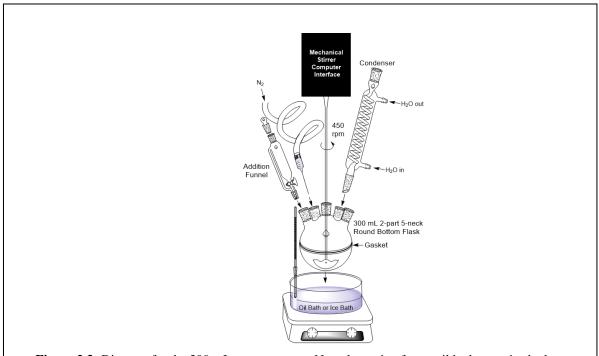


Figure 2.3: Diagram for the 300 mL reactor set-up. Note the option for an oil bath or an ice bath.

#### **Procedure**

- 1. Add 6.055 g of IDA to a solution of 50 mL of 2M NaOH to achieve a final concentration of 0.91M IDA. The beaker was covered with parafilm and left to stir at 350 rpm until fully dissolved, about 10 minutes. The resulting product is 'neutralized IDA'.
- 2. Add the 50 mL solution of neutralized IDA to the reactor and begin the mechanical stirring at 450rpm using the overhead stir bar.

3. Using a 10 mL mechanical pipette, add 6.821 mL of GMA to the addition funnel. Then open the stopcock of the addition funnel slightly to add the reagents to the reactor at a rate of approximately 1 drop per second. The solution will turn from colorless to white upon the addition of GMA.

<u>TIP #3:</u> Angle the tip of the funnel towards the center of the round bottom flask so the reagent is added directly to the center of the reaction, and to avoid losing reagent from rolling down the edges of the flask.

4. After all the GMA has been added to achieve a final concentration of 0.91M corresponding to a 1:1 molar ratio of GMA:IDA, replace the addition funnel with a rubber septum stopper and let the reaction proceed for 1 hour.

<u>NOTE:</u> The mixture will turn from colorless to white after the addition of GMA.

5. Using a 5mL mechanical pipette, neutralize the crude product with 2mL of concentrated HCl.

TIP #3 applies: angle the pipette tip towards the center of the reaction to avoid losing acid on the sides of the round bottom

- 6. Purify the GMA-IDA product by washing.<sup>9</sup>
  - i. Add the crude product along with 300 mL of acetone to a 500 mL separatory funnel. Gently shake the funnel, and release the stopcock to relieve pressure. Repeat this inversion process until there is no longer pressure buildup after inversion. In other words when there is no longer a sound upon of pressure-release.

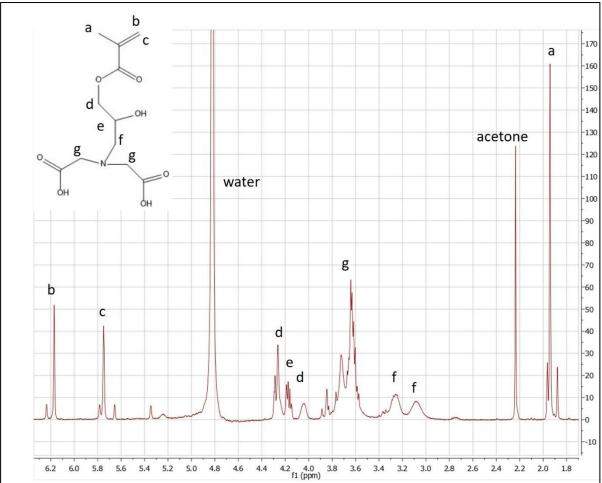
<u>TIP #2:</u> Make sure to be gentle when shaking the separatory funnel as vigorous shaking may cause excess pressure to buildup.

- ii. Secure the funnel using a ring stand and leave it to sit for 5-10 minutes to allow the inorganic and organic layers to separate.
- iii. Remove the stopper of the separatory funnel and open the stopcock slightly to slowly drain the bottom aqueous layer containing the GMA-IDA. Discard the top layer.
- iv. Repeat steps i-iii with 50 mL DI water keeping the aqueous layer and discarding the organic layer.
- v. Repeat steps i-iv three more times plus an additional final wash with 300 mL acetone.
- vi. Dry the product in a vacuum desiccator for 20 minutes at room temperature to remove excess acetone.
- 7. The final product should be a clear, slightly viscous liquid. Store at 4°C until it is needed for the copolymerization in Part III.

<u>TIP #4:</u> Storage conditions should be altered appropriately for light or moisture sensitive monomers, for example to store in an amber vial or over desiccant.

#### Method Validation

To verify the product has the structure presented in Figure 2.4, use proton NMR.



**Figure 2.4:** Proton NMR spectrum of 2-propenoic acid,2-methyl-,3-[bis-(carboxymethyl) amino]-2-hydroxypropyl ester (GMA-IDA) in D<sub>2</sub>O (400 MHZ). Reprinted from Colloids and Surfaces B: Biointerfaces, Vol. 172, Lin, Z., Goddard, J., *Photocurable coatings prepared by emulsion polymerization present chelating properties*, pp 143-151, 2018.

#### 2.5 Part II

## Synthesis of Monomer 3: 4-benzoylphenyl methacrylate (BPM)

#### **Reaction Duration**

20 hours + 3-day total: 0.5 hours for synthesis preparation + 18-hour synthesis + 1.5 hour purification + 3-day dry

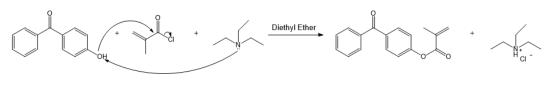


Figure 2.5: Mechanism for the synthesis of monomer 3: 4-benzoylphenyl methacrylate (BPM).

#### Reagents

4-hydroxybenzophenone (98%), diethyl ether, triethyl amine (99.5%), methacryloyl chloride (97%), DI water, sodium hydroxide pellets and magnesium sulfate.

# **Equipment**

The reactor consists of a 300 mL two-part, 5-neck round bottom flask, overhead mechanical stirrer, glass stirring rod and anchor style agitator, reactor stand, battery operated thermometer with clip, ice bath, funnel, nitrogen line and 3 rubber septum stoppers. Other equipment includes 10 mL syringe, 5 mL syringe, 2 sparging needles, stopcock compatible with syringe and needle, parafilm, scissors, aluminum foil, 200 mL graduated cylinder, 10mL mechanical pipette, filter paper and a Buchner funnel.

#### Preparation

1. Assemble the reactor as shown in Figure 2.3, including an addition funnel, nitrogen line and ice bath. Plug the three free necks with rubber septum stoppers.
NOTE: The ice bath is to prevent excess heat in the beginning of the reaction. It is not necessary to keep the ice bath cold throughout the duration of the reaction. If your specific reaction requires a cold ice bath for a long period of time, considering employing an immersion chiller probe. See link above in "Resource Availability."

- <u>TIP #5:</u> make sure the agitator anchor is as close to the bottom of the round bottom flask as possible to ensure the homogenization of 4-hydroxylbenzophenone and diethyl ether.
- Purge the reactor and addition funnel with nitrogen and then plug the reactor and turn the flow off. You do not need a continuous flow of nitrogen throughout the reaction.
- 3. To prepare for air-free collection of methacryloyl chloride
  - i. Cut a 10 mL syringe equipped with a stopcock at the 4 mL mark using scissors. Wrap Parafilm® around the cut end of the syringe to prepare it for an air tight seal with a rubber balloon. Place the mouth of a rubber balloon around the Parafilm® wrapped syringe and seal it as tight as possible with a plastic cable tie.
  - ii. With the stopcock of the syringe open, fill the balloon with nitrogen and close off the stopcock. Connect a sparging needle to the end of the stopcock on the syringe and plunge it into the rubber top of the methacryloyl chloride ensuring that the tip of the needle remains in the air region of the bottle, above the reagent.
  - top of the reagent bottle, feeding the needle all the way down into the reagent. Open the stopcock on the syringe holding the nitrogen filled balloon to allow nitrogen to flow to replace the collected reagent. Use the 5 mL syringe to collect 5 mL of methacryloyl chloride. Flip the

syringe upside down while still in the reagent to rid the vessel of any air bubbles.

<u>TIP #6:</u> When handling methacryloyl chloride, wear gloves (natural rubber, butyl rubber, nitrile rubber, neoprene or polyvinyl chloride (PVC)), protective goggles and a respirator mask and work in the fume hood since this is a volatile and toxic reagent. Methacryloyl is flammable, toxic if inhaled and corrosive.

4. Prepare a 0.1% w/w NaOH solution using the method mentioned in step 2 of *Synthesis Preparation* in Part I.

# Procedure

- Using a 200mL graduated cylinder, add 150mL diethyl ether to the reaction vessel and set the overhead stirring to 450rpm.
  - <u>TIP #7:</u> Diethyl Ether is a peroxide former test your Diethyl Ether every 6 months for peroxides.
- Add 10.32g of 4-hydroxybenzophenone to the reaction vessel and let homogenize for 10 minutes.
- 3. Using a 10mL pipette, charge 7.985mL of triethyl amine into the reaction vessel and then purge the reaction with nitrogen to achieve a 1:1 molar ratio of triethylamine:4-hydroxybenzophenone.
- 4. Cover the reaction vessel in foil to allow the reaction to proceed in a light-free environment since the product is light sensitive.

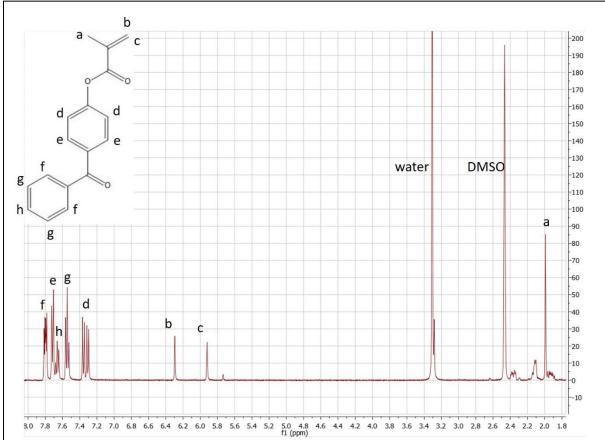
- <u>TIP #8:</u> Wrap the foil in such a way to create an easy peel-back flap that allows for reaction monitoring if necessary.
- 5. Add 25 mL of diethyl ether to the addition funnel.
- To the diethyl ether filled addition funnel, add the previously collected 5 mL methacryloyl chloride.
  - <u>TIP #9:</u> The sparging needle was placed directly in the diethyl ether to add the methacryloyl chloride to ensure homogenization of the reagents.
- 7. Open the stopcock of the addition funnel to allow the reagents to add dropwise to the reaction to achieve a 1:1:1 molar ratio of methacryloyl chloride:triethylamine:4-hydroxybenzophenone. The solution should appear white and liquid. Afterwards, replace the addition funnel with a rubber stopcock and let reaction was proceed in the dark for 18 hours.
  - TIP #3 again applies here.
- 8. After the completion of the reaction, filter off the triethylammonium hydrochloride precipitate using filter paper and a glass funnel.
  - NOTE: The product should be a white liquid, not a yellow solid. If the resulting product is solid, it is most likely there was a significant leak in the reactor causing the diethyl ether fumes to evaporate.
  - <u>TIP #10:</u> Carry out all post-synthesis steps in as low-light an environment as possible to since BPM is a light sensitive reagent. Use foil whenever possible.
- Wash the filtrate with 200 mL DI water and 200 mL 0.1% NaOH following the same extraction procedure mentioned in Part I, until no yellow color is observed in the organic phase.

- 10. Dry the product over MgSO<sub>4</sub>
  - i. Using a metal scooper, add an excess of magnesium sulfate to the product collected in step 9.
  - ii. Swirl the product between additions of magnesium sulfate.
  - iii. Repeat with additions of magnesium sulfate until you see the *snowstorm* effect. This is when the magnesium sulfate is freely flowing and takes a while to settle to the bottom, similar to what is seen in a snow globe.
- 11. Evaporate the diethyl ether from the BPM using a vacuum and Buchner funnel.

  The resulting product should be a white solid.
- 12. Dry the product in a vacuum desiccator for 3 days to collect the BPM and store in a sealed foil-covered vessel in the refrigeration until further use.

# Method Validation

To verify the product has the structure presented in Figure 2.6, use proton NMR.



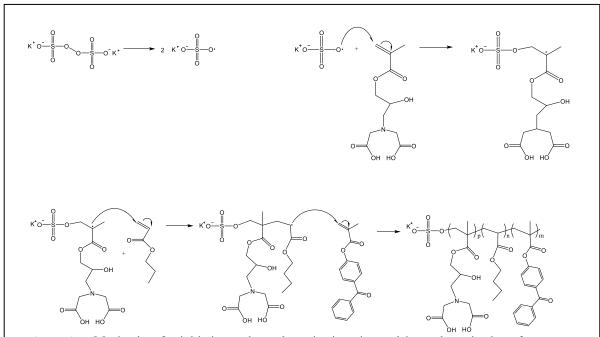
**Figure 2.6:** Proton NMR spectrum of 4-benzoylphenyl methacrylate (BPM) in DMSO-d<sub>6</sub> (400 MHZ). Reprinted from Colloids and Surfaces B: Biointerfaces, Vol. 172, Lin, Z., Goddard, J., *Photocurable coatings prepared by emulsion polymerization present chelating properties*, pp 143-151, 2018, with permission from Elsevier.

# 2.6 Part III

Emulsion Polymerization for the copolymer synthesis (poly(2-propenoic acid,2-methyl-,3-[bis-(carboxymethyl) amino]-2-hydroxypropyl ester-co-n-butyl acrylate-co-4-benzoylphenyl methacrylate) (GMA-IDA-co-BA-co-BPM))

# **Reaction Duration**

27 hours total: 0.5-hour synthesis preparation + 20-hour reaction + 6.5-hour product purification



**Figure 2.7:** Mechanism for initiation and copolymerization via emulsion polymerization of monomers glycidyl methacrylate-iminodiacetic acid (GMA-IDA), butyl acrylate (BA), and 4-benzoylphenyl methacrylate (BPM).

<u>NOTE:</u> This is a random copolymer, i.e. the monomers will add to the chain randomly and in unequal proportions. See Figure 2.8.

# Reagents

Butyl acrylate (BA), potassium persulfate (KPS) (99+%), DI water, methanol, GMA-IDA synthesized from Part I and BPM synthesized from Part II.

# **Equipment**

The reactor consists of a 300 mL two-part, 5-neck round bottom flask, overhead mechanical stirrer, glass stirring rod and anchor style agitator, reactor stand, hot plate, battery operated thermometer with clip, oil bath set to 70°C, condenser, nitrogen line and 4 rubber septum stoppers. Other equipment includes a 150 mL graduated cylinder, 10 mL mechanical pipette, 50 mL beaker, 1 mL mechanical pipette, and regenerated cellulose dialysis membranes.

# Preparation

1. Assemble the reactor as shown in Figure 2.3, including the nitrogen line and oil bath heated to 70°C. Plug the free necks with rubber septum stoppers.

# Procedure

- Add 2.7 g of GMA-IDA and 0.39 g KPS to the reactor along with 126 mL of DI water to achieve a 10:1 molar ratio of GMA-IDA monomer: KPS initiator.
   Homogenize the reagents at a stir rate of 450 rpm.
- 2. Dissolve 2.4g of BPM in 10.8 mL of BA and charge into the reaction vessel to yield a total monomer ratio of 7:1:1 BA:GMA-IDA:BPM.
- 3. Start the nitrogen flow to the reactor and bring the oil bath up to 70°C. Cover the reactor with foil and the let it proceed for 20 hours in the dark.

  \*TIP #8 again applies here.
- The crude product should be a white liquid. Centrifuge the crude product at 3000 x g for 15 minutes to remove polymer sediments. Repeat twice.
- 5. Treat the supernatant with dialysis to remove the unreacted reagents:
  - i. To remove the unreacted GMA-IDA"
    - a. Using scissors, cut 20kDa dialysis membrane tubes according to the required volume per length listed on the packaging of the dialysis tubes.
      - TIP #11: Leave an extra 5-6 cm of tubing to account for the clips that will be placed on either end.
    - b. Soak the tube in DI water for 10 minutes.
    - c. Fold over one end of the tube and secure it with a clip and add the copolymer to the tube. Clip the other end.

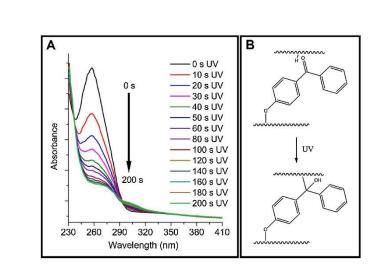
- <u>TIP #12:</u> Put the tube in a small beaker clip side down to catch spills. The tubing is quite flexible so this prevents likely loss of polymer.
- d. Fill a 1 L beaker with DI water and place a magnetic stir bar at the bottom. Place the filled dialysis tube in the DI water, put the beaker on a magnetic stir plate and begin stirring at 350 rpm.
- e. Let the dialysis continue for 3 hours.
- ii. To remove the unreacted BA and BPM:
  - a. Repeat step i, a-e except substitute DI water for methanol.
- 6. Centrifuge the purified retentate twice more at 3000 gs for 15 minutes and collect the purified polymer supernatant and store in the refrigerator.

# Method Validation

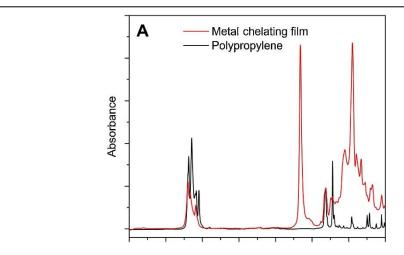
To verify the product has the structure presented in Figure 2.8, use proton NMR. aromatic protons **DMSO** aromatic protons ethylene 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 f1 (ppm) 3.5 3.0 2.5 2.0 1.5 Figure 2.8: Proton NMR spectrum of GMA-IDA-co-BA-co-BPM polymer collected in DMSO-d<sub>6</sub> (130 °C, 600 MHZ). Reprinted from Colloids and Surfaces B: Biointerfaces, Vol. 172, Lin, Z.,

Goddard, J., Photocurable coatings prepared by emulsion polymerization present chelating properties, pp 143-151, 2018, with permission from Elsevier.

To verify that the copolymer is curable via exposure to UV irradiation, monitor the absorption spectrum at 270-290 nm as the coating is being exposed to UV-light (365 nm). The absorption band at 270 nm should decrease as a result of successful benzophenone crosslinking as shown in Figure 2.9. To verify the copolymer contained poly(n-butyl acrylate) and GMA functionalized with IDA ligands, take ATR-FTIR and absorbance spectra of polypropylene coated with the copolymer compared to bare polypropylene as the control as shown in Figure 2.9. Bare polypropylene should show absorption bands at 3000-2800 cm<sup>-1</sup> for the C-H stretch and at 1450 cm<sup>-1</sup> and 1370 cm<sup>-1</sup> <sup>1</sup> for the C-H bend. For the copolymer film, absorption band at 3000-2800 cm<sup>-1</sup> for the C-H stretch and a strong absorption band at 1710 cm<sup>-1</sup> for the carbonyl stretch and bands at 1260-1160 cm<sup>-1</sup> for a ether stretch indicate poly(n-butyl acrylate) is present while a small shoulder at shoulder at 1620 cm<sup>-1</sup> indicate the presence of IDA ligands.<sup>10</sup>



**Figure 2.9:** Absorption spectra of benzophenone during UV-curing (A) and the mechanism of benzophenone before and after UV-curing (B). Reprinted from Colloids and Surfaces B: Biointerfaces, Vol. 172, Lin, Z., Goddard, J., Photocurable coatings prepared by emulsion polymerization present chelating properties, pp 143-151, 2018, with permission from Elsevier.



**Figure 2.10:** ATR-FTIR spectra for bare polypropylene and the copolymer film. Reprinted from Colloids and Surfaces B: Biointerfaces, Vol. 172, Lin, Z., Goddard, J., *Photocurable coatings prepared by emulsion polymerization present chelating properties*, pp 143-151, 2018, with permission from Elsevier.

## 2.7 Conclusion

The above method provides a detailed synthesis of a functional terpolymer via emulsion polymerization including details for the reactor set-up to ensure a controlled reaction environment. Thus, these methods provide a foundation and synthesis 'tips and tricks' that can be applied and adapted to generate copolymers with different functionalities utilizing emulsion polymerization methods. For example, thermocuring monomers, antimicrobial or non-fouling functional ligands, bio-based environmentally sustainable monomers, or biocompatible monomers with applications in food packaging, food safety, biomedical implants, or functional polymer coatings.

# Acknowledgements

This project was supported in part by Agriculture and Food Research Initiative grant no. 2019-38420-28975 from the U.S. Department of Agriculture, National Institute of Food and Agriculture, and in part by Hatch under Accession Number 1014103. This work made use of the Cornell Center for Materials Research (CCMR) Facilities supported by the GS5>National Science Foundation GS5>under Award Number DMR-1719875. NMR analysis was conducted in the Cornell NMR facility with the technical assistance from Dr. Ivan Keresztes and Anthony Condo. We also thank Autumn Rudlong and Hannah Zurier for assistance with the experiments.

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# CHAPTER 3

## **CONCLUSIONS & FUTURUE DIRECTIONS**

# 3.1 Public Engagement – Future Directions

Science energizes some, while it intimidates to others. To some, chemistry class is the most exciting part of the day, while others dread balancing equations or understanding the mole fractions. Whatever emotion people might associate with science in general, there persists a general appetite to understand. Climate change, nutrition, and public health are common concerns and thus popular subjects to talk or post on social media about. The danger is when facts about topics whose solutions rely on policy and public opinion are miscommunicated and false knowledge is spread. Proper science communication, where experts engage with the public, is the most direct way to address this issue. Training scientists and encouraging them to bridge the knowledge gap is imperative.

Thus, the development of a versatile and adaptable program is one way to facilitate engagement between experts and the general public. In fact, the value in the program lies within its flexibility so that it may be conformed to different settings with varying spaces, budgets and audiences and educators. The food science education program presented in Chapter 1 of this thesis is one example of such program, but its reproducibility is the key to making future impacts. If programs can be put into a format that encourages reproduction, science education will become more mobile. The specific instructions and activity outlines provided lay the groundwork for science

communication, but it also motivates the program facilitators or the activity instructors to continuously engage in science communication now and in the future.

Young minds in particular are important to engage in the sciences. It has been shown that children with early exposure to science education are more scientifically literate. Thus efforts to educating young minds in the sciences can also aid in the continuation of proper science communication. Educating young minds in food science & technology is specifically important if we are to tackle some of the aforementioned global issues such as agricultural sustainability, public health, food security to name a few. Although, students are not generally introduced to food science as a discipline until the university level as they are not offered in elementary or middle school curricula. Providing students with more career opportunities or awareness creates space for a student to be inspired and motivated by prospective future options. This excitement is another source of encouragement for success and comprehension in the sciences which in turn fosters the development of good science communication skills. Furthermore, Food Science & Technology is an area of study that is widely growing at the university level. In fact, employment for jobs that require a food science degree is projected to grow 7% from 2018 to 2028 according to the U.S. Bureau of Labor Statistics.<sup>2</sup> This growth can be attributed to increasing research in the field, which again, is aimed towards overcoming hurdles in agricultural production and environmental challenges. Thus, future directions for science communication and science education in public engagement should be aimed at, but not limited to, the development of young scientific minds as they are our future.

## 3.2 General Methods – Future Directions

As previously discussed, general methods for emulsion copolymerization are extremely helpful in expediting research in the field of functional terpolymers. Here I will present an example of how I might alter the method above, using the same principals but achieving different goals.

Food waste is a serious environmental concern as 40% of food is wasted in the U.S. annually<sup>3,4</sup> resulting in unnecessary carbon emissions. In 2013, the FAO consolidated carbon emission data of global food waste alone alongside top country carbon emissions, food waste ranking third below the U.S. and China.<sup>5</sup> Active packaging modifies the environment of packaged foods and provides an excellent solution to food waste as it can extend the shelf life of food products by preventing microbial spoilage or chemical degradation such as lipid oxidation.<sup>4</sup> Furthermore, active packaging that immobilizes the preservatives simultaneously addresses consumer demands and the clean label trend<sup>6</sup> since the preservatives do not migrate into the food but stay chemically tethered to the packaging material. There are many ways to prepare and implement active packaging, one of which is photografting where UV light (315-400nm) is used to generate free radicals on polymer chains which then provide a reactive cite for curing onto a surface.<sup>7</sup>

**Scheme 3.1:** UV-cure of 4-benzoylphenyl methacrylate onto a polypropylene backbone via a hydrogen abstraction mechanism

The emulsion polymerization protocol presented in Chapter 1 yields the type of copolymer film described above. Poly(2-propenoic acid,2-methyl-,3-[bis-(carboxymethyl) amino]-2-hydroxypropyl ester-*co-n*-butyl acrylate-co-4benzoylphenyl methacrylate) (GMA-IDA-co-BA-co-BPM) presents metal-chelating capabilities via the GMA-IDA monomer which have been shown to prevent lipid oxidation in foods<sup>8,9</sup> and thus prolongs shelf life. In addition, the carbonyl group on the BPM (4-benzoyphenyl methacrylate) monomer when exposed to UV light at 365nm generates a radical that abstracts a hydrogen from an alkyl group on a polypropylene surface, 10 a common material for food packaging. 7 This mechanism can be seen in Scheme 3.1. UV curable coatings are advantageous because they do not require high amounts of energy, they cure in 180 seconds<sup>10</sup> and release less volatile organic compound emissions.<sup>11</sup>

While the metal-chelating and photocurable functionalities of GMA-IDA-co-BA-co-BPM provide some environmentally greener solutions, the synthesis of the photocurable monomer, requires the use of methacryloyl chloride, an environmentally toxic and corrosive chemical.<sup>12</sup> Thus, to further improve this environmental sustainability of this synthesis, the protocol presented in Chapter 1 may be adjusted to

include a synthesis of a photocurable moiety using less harsh reagents. One approach to this is to replace BPM with a photocurable bio-based monomer.

Itaconic acid (IA), structure shown in Scheme 3.2, is a renewable, unsaturated dicarboxylic acid that has attracted attention for research in polyester synthesis. <sup>13</sup> In fact, this fully bio-based monomer is recommended by the U.S. Department of Energy as one of the top 12 bio-based chemicals. <sup>14</sup> In order to incorporate itaconic acid into this copolymer as the UV-curable ligand, the structure must be altered since it is a difunctional acid, meaning it is prone to crosslinking. <sup>15</sup> Thus, an itaconic acid monoester,  $\beta$ -ethylester itaconic acid (IM), can be synthesized in two steps according to a reported procedure. <sup>15</sup>

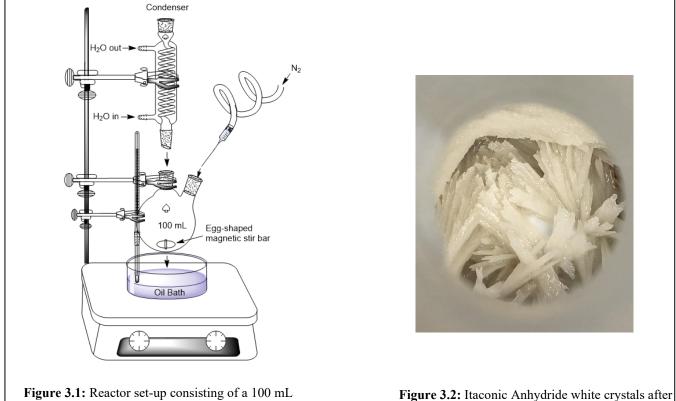
# Materials

Itaconic acid, sulfuric acid, toluene, ethanol, glycidyl methacrylate, triphenylphosphine, 4-methoxy phenol, hydrochloric acid, sodium hydroxide, cresol red indicator and thymol blue indicator.

# Procedure

The reactor set-up used for the following synthesis consists of a 100mL two-neck round bottom flask in an oil bath with an egg-shaped magnetic stir bar, condenser, and nitrogen line as shown in Figure 3.1. Briefly, Itaconic Anhydride was first synthesized by reacting 1 eq. of IA with 1.0 eq. acetic anhydride with 0.08 wt% sulfuric acid in toluene at 50° under reflux and stirring for 3 hours. The acetic acid was evaporated using a rotary evaporator and the white crystalline product was obtained as

seen in Figure 3.2. Second, 1.0 eq. of itaconic anhydride was further reacted with 1.0 eq. ethanol in toluene  $50^{\circ}$  under reflux and stirring for 16 hours to yield  $\beta$ -ethylester itaconic acid (IM), as shown in Scheme 3.1. Next, to



**Figure 3.1:** Reactor set-up consisting of a 100 mL two-neck round bottom flask, magnetic stir bar, condenser, nitrogen line and oil bath.

**Figure 3.2:** Itaconic Anhydride white crystals after evaporating acetic acid via rotary evaporation.

prepare the monomer for copolymerization, GMA (glycidyl methacrylate) can be functionalized with IM via an epoxy ring-opening esterification according to a reported procedure<sup>16</sup> with modifications. Starting without removing the IM from the round bottom flask from the previous step, the reaction was purged with nitrogen and the temperature of the oil bath was raised to 95°C. For 1.0 eq. of IM, 1.0 eq. of GMA with 0.5 wt% catalyst triphenylphosphine and 0.1 wt%

# a) Itaconic Anhydride Synthesis

#### Itaconic Acid

# Product: Itaconic Anhydride

# Byproduct: Acetic Acid Byproduct: Acetic Acid

## b) β-Ethylester Itaconic Acid Synthesis

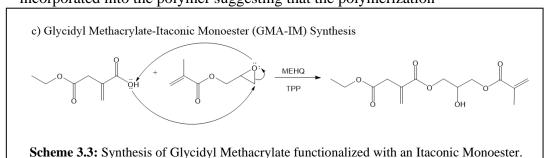
## Product: Itaconic Monoester

**Scheme 3.2:** Synthesis of Itaconic Anhydride (a) followed by synthesis of  $\beta$ -ethylester itaconic acid (b).

radical inhibitor 4-methoxy phenol was added. After 30 minutes, the temperature was raised 110-120°C and the reaction was left to run until the epoxy value reached 0. The epoxy value was determined via titrations with hydrochloric acid and sodium hydroxide using cresol red and thymol blue indicators. The reaction was cooled to 30°C. The reaction is shown in Scheme 3.3. The product, GMA-IM monomer, can be characterized using FTIR looking for characteristic bands at 3468cm<sup>-1</sup> for the hydroxyl groups, 2958 cm<sup>-1</sup> for alkyl groups, 1720 cm<sup>-1</sup> for carbonyl groups and 814cm<sup>-1</sup> and 1637 cm<sup>-1</sup> for terminal alkene groups. The GMA-IM monomer can then be copolymerized with GMA-IDA and BA via emulsion polymerization following the protocol presented in Chapter 1.

# Discussion

There are some potential problems that could arise when swapping out a monomer for another, in this case replacing BPM with GMA-IM. This copolymer film is intended to be a random copolymer, in that the monomers add polymer chain in no particular order. Previously, the monomers were added in a 7:1:1 ratio BA:GMA-IDA:BPM which will be repeated, however the proton NMR spectrum of GMA-IDA-co-BA-co-BPM presented in Figure 2.8 shows a monomer ratio of 40:1:10 once incorporated into the polymer suggesting that the polymerization



rate could be higher for the addition of BPM than GMA-IDA. If the photocurable monomer, IM, must be present in 10x the amount of the metal-chelating monomer, GMA-IDA, to result in the same mechanical properties, such as curing ability and hydrophobicity, then the initial mole ratios of monomer added might need to be altered since the polymerization site for both monomers are GMA it is likely the polymerization will not strongly favor the addition of one over the other. However, there are two terminal alkenes viable for polymerization on the GMA-IM monomer so a challenge will be getting the polymerization to select for, or favor, the GMA alkene since the alkene on the IM is intended for photocuring to the polypropylene backbone. Another possibility for variation in reactivity of the GMA-IM monomer is that it has been shown

for the terminal alkene can isomerize at temperatures around 180°C resulting in an internal alkene. However, the copolymerization occurs at 95°C which is well below the threshold. Finally, since the photografting site on GMA-IM is at the alkene (in contrast to the carbonyl on the BPM monomer) and thus will proceed via a different mechanism. The use of the photoinitiator, Irgacure 184 will be employed and thus UV light wavelengths (100-400nm) might need to be adjusted as well as exposure time, and the curing mechanism (i.e. whether it proceeds via hydrogen abstraction, electron transfer or cleavage) will need to be determined.

## 3.3 Final Conclusions

Scientific advancement, the foundation of world improvement, cannot happen if knowledge is not communicated effectively. However, science communication is a skill that takes time to learn and develop. This thesis provides two approaches to practice and implement science communication in academia. Whether it be in providing more detailed protocols to make methods more accessible and easily altered, or to provide better training opportunities for science graduate students to interact and educate the public, integrating better science communication is extremely important. It is therefore imperative that work continue towards bettering the science communication skills of all scientists. Moreover, it is critical that the public develop better science communication skills as well. Those who are scientifically literate can and should encourage non-experts to listen to scientific voices. The public who recognizes the limits of their science knowledge will be more open to listening and learning science concepts before spreading false information. In fact, it is the responsibility of those who are literate in

the sciences to learn how to communicate effectively because that will in-turn result in a better-informed public.

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